Supporting Information

for

Iron-Catalyzed Alkylation of Alkenyl Grignard Reagents

Gérard Cahiez,* Christophe Duplais and Alban Moyeux

Laboratoire de Synthèse Organique Sélective et de Chimie Organométallique (SOSCO), *UMR 8123 CNRS-UCP-ESCOM, 5 mail Gay Lussac, Neuville sur Oise 95031 Cergy-Pontoise Cedex, France*

gerard.cahiez@u-cergy.fr

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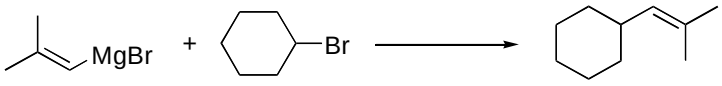
I/ Influence of the Nature of the Catalytic System

It should be noted that the reaction can also be performed in the presence of 1.6 equivalents of TMEDA (RMgBr/TMEDA = 1:1) according to the procedure recently described by Nakamura[1] for the iron-catalyzed aryl-alkyl coupling. (Table S1, entry 2). However, this procedure is less efficient since the yield is clearly lower and the amount of amine is higher (Entries 1 and 2).

Table S1



Entry	RMgX Number of equiv.	Conditions[a]	Yield (%)
1	1.5	5% [$Fe(acac)_3$/TMEDA/HMTA] (1:2:1)	75
2	1.6	5% $FeCl_3$, 160% TMEDA[b]	50

[a] All reactions were performed on a 5 mmol scale in THF at 0°C. [b] RMgBr/TMEDA = 1:1, the amine has to be mixed with the Grignard prior to the addition.

II/ General

All reactions were carried out under a nitrogen atmosphere. Starting materials were purchased from commercial sources and used without any further purification. Anhydrous THF was purchased from Carlo Erba. All products were purified by distillation. Purity of isolated compounds ranges from 99 to 96% as determined by GC analysis (capillary column HP-5MS; 30m x 0.25mm x 0.25µm). All compounds give satisfactory centesimal analysis. The analytical data for the known compounds were found to match with the literature data.

^1H and ^{13}C-NMR spectra were recorded on a JEOL ECX-400 spectrometer. Mass spectra were obtained on a Hewlett-Packard HP 5973 spectrometer via a GC/MS coupling with a Hewlett-Packard HP 6890 chromatograph equipped wia th a capillary column HP-5MS. Ionisation was obtained by electronic impact (EI, 70 eV). Mass spectra are reported as *m/z*.

The stereochemical purity of the products was determined by gas chromatography analysis. In the case of alkenylmagnesium bromides, the stereochemical purity was determined by iodolysis then GC analysis of the resulting mixture of (*Z*)- and (*E*)-alkenyl iodides.

The Grignard solutions were titrated according to the procedure described by Watson [2].

III/ Preparation of Alkenyl Halides

1/ Synthesis of 1-Bromo-2-methylpropene

This compound is synthesized according to the procedure described by Farrell and Bachman.[3]

2/ Synthesis of Stereochemically pure (*Z*)-1-Bromopropene

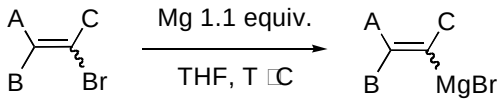
This compound is synthesized according to the procedure described by Fuller and Walker.[4]

IV/ Preparation of Alkenylmagnesium Bromide from Alkenyl Bromide and Magnesium.

1/ General Procedure

The alkenyl Grignard reagents used for performing the experiments described in Table 1 were prepared from commercial mixtures of (*Z*)- and (*E*)-alkenylbromides according to the procedure reported by H. Normant[5]. The proportion of (*Z*)- and (*E*)-isomers and the temperature employed to prepare the Grignard are given in Table S2.

Table S2



Entry	A	B	C	Alkenyl bromide E/Z Ratio	Alkenylmagnesium bromide E/Z Ratio	T °C
1	Me	H	H	80/20	86/14	10-15 °C
2	Me	H	Me	83/17	87/13	50 °C
3	Ph	H	H	88/12	78/22	0 °C

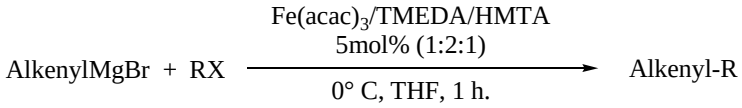
It is well known that the reaction of magnesium with alkenyl halides takes place with a partial isomerization of the double bond.[6] However, it is possible to limit the isomerization by working, as far as possible, at a low temperature.

2/ Stereospecific Preparation of (*Z*)-Propenylmagnesium Bromide

Under stirring, 2 to 3 drops of 1,2-dibromoethane were added to magnesium (1.1 equiv., 66 mmol, 1.60 g) in THF (5 mL). Then, a solution of (*Z*)-1-bromopropene (*Z* > 99%, 1M solution in

THF, 60 mmol, 60 mL) was introduced dropwise at 7 °C. Stirring was continued for 4 h at room temperature. (*Z*)-1-Propenylmagnesium bromide was obtained in 70% yield (Z/E = 97:3).

IV/ Iron-Catalyzed Cross-Coupling Reaction: General Procedure.



A dry 250 mL flask, equipped with a mechanical stirrer and a septum, was charged with THF (15 mL), alkyl halide (25 mmol), $Fe(acac)_3$ (443 mg, 1.25 mmol, 5 mol%), TMEDA (290 mg, 2.5 mmol, 10 mol%) and HMTA[a] (175 mg, 1.25 mmol, 5 mol%). The reaction mixture was cooled to 0°C then the alkenylmagnesium bromide (37.5 mmol), as a solution in THF, was added dropwise for 1 h. After completion of the addition, the reaction mixture was stirred for an additional 30 min, then quenched with a 1M aqueous HCl solution (100 mL). The aqueous phase was extracted with petroleum ether (3 x 30 mL), then the combined organic layers were dried with $MgSO_4$ and concentrated *in vacuo* or distilled under atmospheric pressure, according to the boiling point of the product. The crude residue was purified by distillation at a reduced pressure to afford the coupling product as a colorless oil (55-84%).

[a]*HMTA= hexamethylmethylentetramine*

V/ Spectral Data

3-Methyl-1-nonene (entry 1)

Colorless oil. b.p. = 43 °C /10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.77 (t, J = 6.87 Hz, 3H), 0.82 (d, J = 6.87 Hz, 3H), 1.17 (m, 10H), 2.01 (m, 1H), 4.85 (m, 2H), 5.62 (m, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.13 (CH3), 17.93 (CH3), 22.70 (CH2), 28.95 (CH2), 29.22 (CH2), 29.36 (CH2), 29.55 (CH2), 29.65 (CH2), 31.93 (CH2), 32.62 (CH2), 124.51 (CH), 131.70 CH).

Microanalysis : Calcd. C = 85.63 H = 14.37; Found : C = 85.39 H = 14.61

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 126

4-Methyl-2-decene (entry 2) E/Z : 85/15

Colorless oil. b.p. = 59 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.77 (t, J = 6.87 Hz, 3H), 0.82 (d, J = 6.87 Hz, 3H), 1.17 (m, 10H), 1.55 (dd, J = 6.87 Hz and J = 1.83 Hz, 0.15 x 3H), 1.57 (dd, J = 6.87 Hz and J = 1.83 Hz, 0.85 x 3H), 1.95 (m, 0.85 x 1H), 2.35 (m, 0.15 x 1H), 5.2 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 12.96 (0.15 x CH3), 14.21 (0.85 x CH3), 18.04 (CH3), 20.91 (CH3), 22.78 (CH2), 27.41 (CH2), 29.57 (CH2), 31.21 (0.15 x CH), 32.01 (0.85 x CH), 36.79 (CH2), 37.31 (0.85 x CH2), 37.55 (0.15 x CH2), 122.00 (0.15 x CH), 122.67 (0.85 x CH), 137.43 (0.15 x CH), 137.85 (0.85 x CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.96 H = 14.04

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 154

3,4-Dimethyl-2-decene (entry 3) E/Z : 85/15

Colorless oil. b.p. = 88 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.80 (t, J = 7.33 Hz, 3H), 0.85 (t, J = 6.87 Hz, 3H), 1.18 (m, 10H), 1.47 (m, 6H), 1.98 (m, 0.15 x 1H), 2.55 (sextuplet, J = 7.79 Hz, 0.85 x 1H), 5.11 (q, J = 5.95 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 12.79 (CH3), 14.11 (CH3), 17.94 (CH3), 19.07 (CH3), 22.69 (CH2), 27.67 (CH2), 29.55 (CH2), 31.91 (CH2), 33.29 (CH2), 34.83 (CH), 118.53 (CH), 140.04 (Cq).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.28 H = 14.72

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 168

2,4-Dimethyl-2-decene (entry 4)

Colorless oil. b.p. = 92 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.80 (t, J = 6.87 Hz, 3H), 0.80 (t, J = 6.87 Hz, 3H), 1.16 (m, 10 H), 1.52 (s, 3H), 1.59 (d, J = 1.37 Hz, 3H), 2.2 (m, 1H), 4.78 (dt, J = 9,16 Hz and J = 1.37 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.11 (CH3), 17.90 (CH3), 21.29 (CH3), 22.69 (CH2), 25.77 (CH3), 27.48 (CH2), 29.54 (CH2), 31.95 (CH2), 32.39 (CH), 37.88 (CH2), 129.49 (CH), 131.69 (CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.52 H = 14.48

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 168

3-Cyclohexyl-2-methyl-2-propene[8] (entry 5)

Colorless oil. b.p. = 94 °C/100 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.0 (m, 2H), 1.26 (m, 2H), 1.55-1.68 (m, 5H), 1.59 (d, J = 0.92 Hz, 3H), 1.65 (d, J = 1.37 Hz, 3H), 2.10 (m, 1H), 4.92 (m, 1H)

^{13}C-NMR (100 MHz, $CDCl_3$) δ 17.81 (CH2), 18.05 (CH2), 25.77 (CH2), 26.00 (CH2), 26.13 (CH3), 26.37 (CH2), 33.39 (CH3), 33.63 (CH2), 37.04 (CH2), 37.28 (CH), 131.19 (CH), 131.43 (Cq).

Microanalysis: Calcd. C = 86.88 H = 13.12; Found : C = 86.69 H = 13.31

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 138

3-Methyl-1-phenyl-1-pentene[9] (entry 8) E/Z : 76/24

Colorless oil. b.p. = 72 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 7.33 Hz, 0.24 x 3H), 0.92 (t, J = 7.33 Hz, 0.76 x 3H), 1.04 (d, J = 6.87 Hz, 0.24 x 3H), 1.09 (d, J = 6.41 Hz, 0.76 x 3H), 1.35 (quint., J = 7.33 Hz, 0.24 x 2H), 1.40 (quint., J = 7.33Hz, 0.76 x 2H), 2.2 (m, 0.76 x 1H), 2.67 (m, 0.24 x 1H), 5.44 (dd, $^{3}J_{cis}$ = 11.45 Hz and J = 10.53 Hz, 0.24x 1H), 6.11 (dd, $^{3}J_{trans}$ = 16,03 Hz and J = 7.79 Hz, 0.76 x 1H), 6.35 (m, 2H), 7.28-7.36 (m, 5H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 11.83 (CH3), 20.19 (0.76 x CH2), 20.65 (0.24 x CH2), 29.78 (CH2), 30.33 (CH3), 33.76 (CH), 38.9 (CH), 125.92 (4x CHarom), 126.33 (CHarom), 126.71 (2x CHarom), 127.51 (CH), 128.09 (2x CHarom), 128.41 (CH), 128.60 (CHarom), 136.74 (CH), 137.95 (CH).

Microanalysis: Calcd. C = 89.94 H = 10.06; Found : C = 90.12 H = 9.88

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 160

1-Dodecene (entry 9)

Colorless oil. b.p. = 80 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.87 (t, J = 6.87 Hz, 3H), 1.34 (m, 16H), 2.02 (q, J = 7.33 Hz, 2H), 4.91 (dd, $^{3}J_{cis}$ = 10.07 Hz and J_{gem} = 1.37 Hz, 1H), 4.98 (m,1H), 5.8 (m, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.12 (CH3), 22.69 (CH2), 28.95 (CH2), 29.16 (CH2), 29.35 (CH2), 29.51 (CH2), 29.62 (2x CH2), 31.91 (CH2), 33.83 (CH2), 114.06 (CH), 139.27 (CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.83 H = 14.17

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 182

2-Tridecene[8] (entry 10)

Colorless oil. b.p. = 96 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.87 (t, J = 6.87 Hz, 3H), 1.28 (m, 16H), 1.60 (d, J = 5.95 Hz, 0.15 x 3H), 1.62 (m, 0.85 x 3H), 2.01 (q, J = 6.87 Hz, 2H), 5.39 (m, 2H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.10 (CH3), 17.90 (CH3), 22.69 (CH2), 26.84 (0.15 x CH2), 29.21 (CH2), 29.36 (2 x CH2), 29.56 (2 x CH2), 29.65 (CH2), 31.93 (CH2), 32.62 (0.85 x CH2), 123.58 (0.15 x CH), 124.51 (0.85 x CH), 130.91 (0.15 x CH), 131.67 (0.85 x CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.42 H = 14.58

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 182

3-Methyl-2-tridecene (entry 11) Z/E : 86/14

Colorless oil. b.p. = 68 °C/4 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 6.41 Hz, 3H), 1.24 (m, 16H), 1.54 (dd, J = 6.41 Hz and J = 1.37 Hz, 3H), 1.64 (t, J = 1.37 Hz, 3H), 1.80 (t, J = 7.33 Hz, 2H), 5.16 (m, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 13.21 (CH3), 14.12 (CH3), 22.69 (CH2), 23.38 (CH3), 27.80 (CH2), 29.36 (CH2), 29.65 (2 x CH2), 29.70 (2 x CH2), 31.36 (CH2), 31.91 (CH2), 118.61 (0.84 x CH), 136.17 (0.16 x CH), 136.17 (0.16 x CH), 136.46 (0.84 x CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.81 H = 14.19

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 196

2-Methyl-2-undecene (entry 12)

Colorless oil. b.p. = 87 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 6.87 Hz, 3H), 1.25 (m, 12H), 1.58 (s, 3H), 1.67 (d, J = 0.92 Hz, 3H), 1.93 (q., J = 6.87 Hz, 2H), 5.10 (m, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.11 (CH3), 17.66 (CH3), 22.69 (CH2), 25.73 (CH3), 28.05 (CH2), 29.35 (2x CH2), 29.57 (CH2), 29.91 (CH2), 31.91 (CH2), 124.97 (CH), 131.10 (CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.37 H = 14.63

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 168

Ethyl 8-methyl-7-nonenoate (entry 13)

Colorless oil.

purification : flash chromatography (silica gel), petroleum ether / diethyl ether : 99 / 1.

^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.18 (t, J = 6.87 Hz, 3H), 1.25 (m, 4H), 1.52 (s, 3H), 1.54 (quint., J = 7.33 Hz, 2H), 1.61 (d, J = 0.92 Hz, 3H), 1.90 (m, 2H), 2.20 (t, J = 7.33 Hz, 2H), 4.04 (q, J = 6.87 Hz, 2H), 5.03 (m, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.20 (CH3), 17.62 (CH3), 24.89 (CH2), 25.67 (CH3), 27.78 (CH2), 28.76 (CH2), 29.46 (CH2), 34.33 (CH2), 60.10 (CH2), 124.53 (CH), 131.37 (Cq), 173.84 (Cq).

Microanalysis: Calcd. C = 72.68 H = 11.18; Found : C = 72.51 H = 11.02

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 198

9-Methyl-8-decenenitrile (entry 14)

Colorless oil.

purification : flash chromatography (silica gel), petroleum ether / diethyl ether : 99 / 1.

^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.25 (m, 4H), 1.38 (m, 2H), 1.52 (d, J = Hz, 3H), 1.58 (quint., J = 7.33 Hz, 2H), 1.62 (d, J = 0.92 Hz, 3H), 1.9 (m, 2H), 2.26 (t, J = 7.33 Hz, 2H), 5.02 (tq, J = 6.87 Hz and J = 1.37 Hz, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 17.04 (CH3), 17.59 (CH2), 25.27 (CH3), 25.65 (CH2), 27.73 (CH2), 28.30 (CH2), 28.51 (CH2), 29.42 (CH2), 119.80 (Cq), 124.34 (CH), 131.47 (Cq).

Microanalysis: Calcd. C = 79.94 H = 11.59 N = 8.47; Found : C = 80.23 H = 11.68 N = 8.09

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 165

(*Z*)-4-Methyl-2-decene (Scheme 1)

Colorless oil. b.p. = 59 °C/10 Torr

^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.77 (t, J = 6.87 Hz, 3H), 0.82 (d, J = 6.87 Hz, 3H), 1.17 (m, 10H), 1.5 (dd, J = 6.87 Hz and J = 1.83 Hz, 3H), 2.35 (m, 1H), 5.03 (m, 1H), 5.2 (m, 1H).

^{13}C-NMR (100 MHz, $CDCl_3$) δ 12.96 (CH3), 14.11 (CH3), 21.13 (CH3), 22.68 (CH2), 27.43 (CH2), 29.49 (CH2), 31.21 (CH2), 31.92 (CH), 37.55 (CH2), 122.00 (CH), 137.43 (CH).

Microanalysis: Calcd. C = 85.63 H = 14.37; Found : C = 85.96 H = 14.04

MS (EI, 70 eV) *m/z* $[M+H]^+$ = 154

VI/ References

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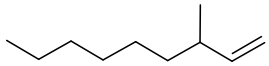

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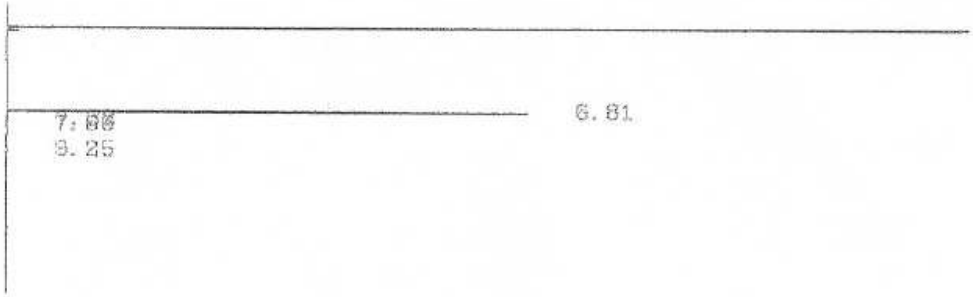


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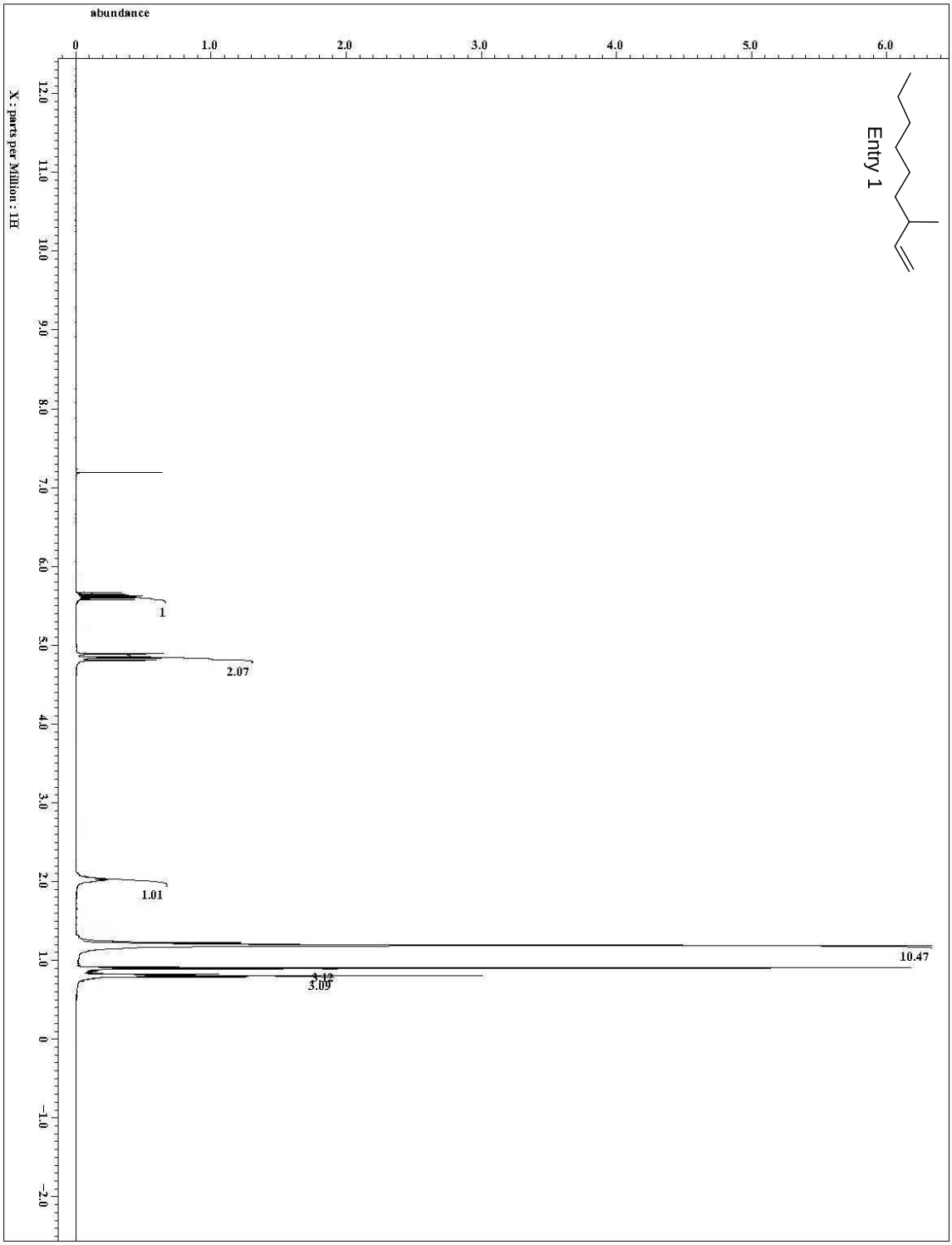
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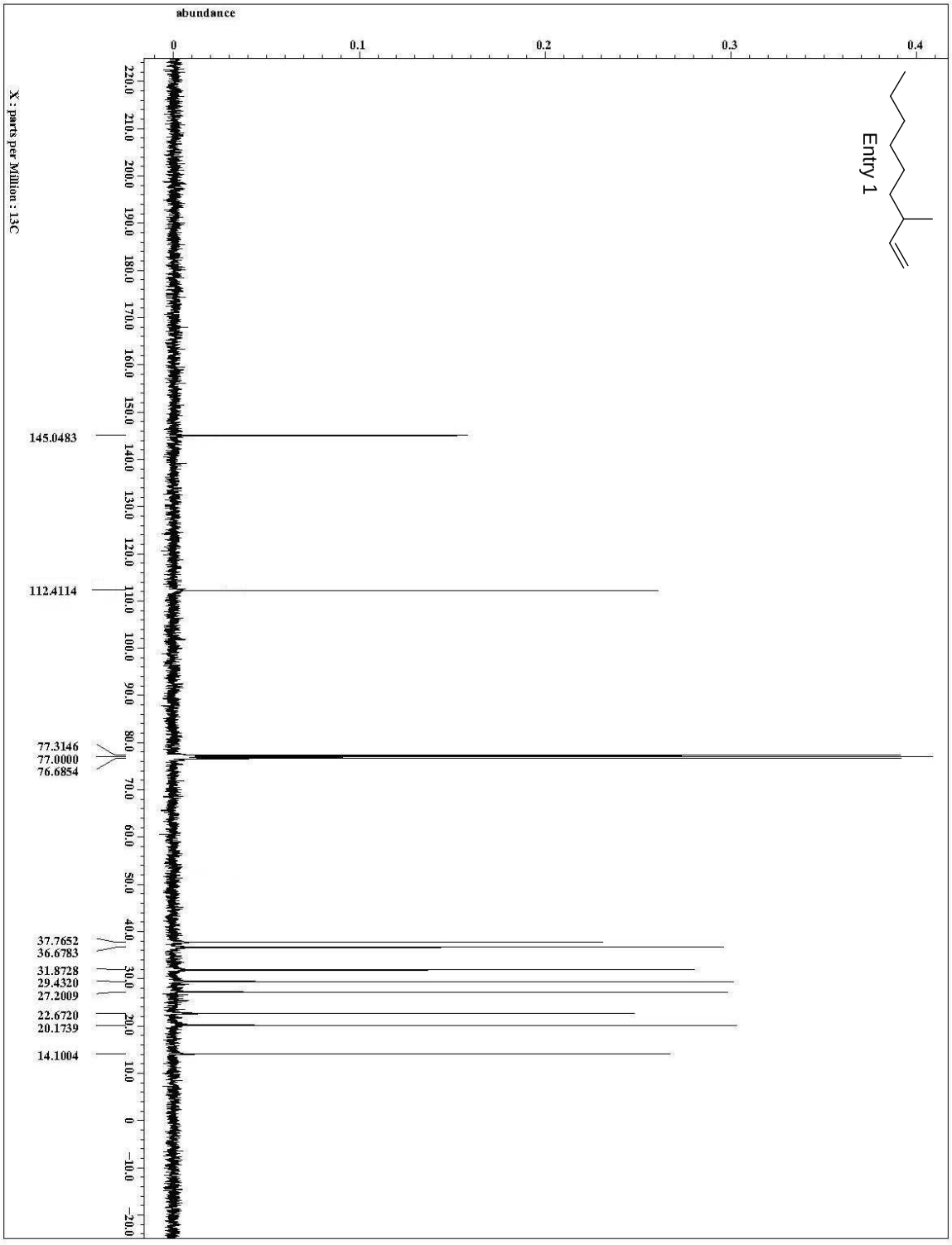
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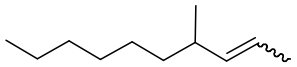

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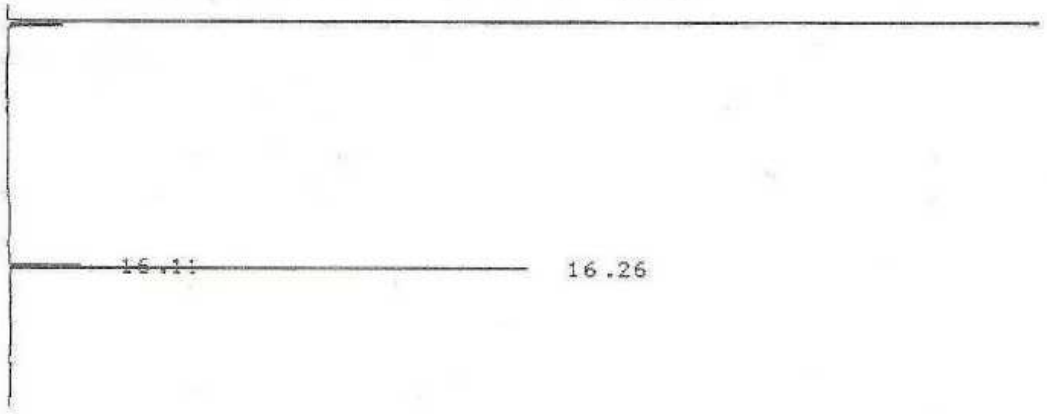
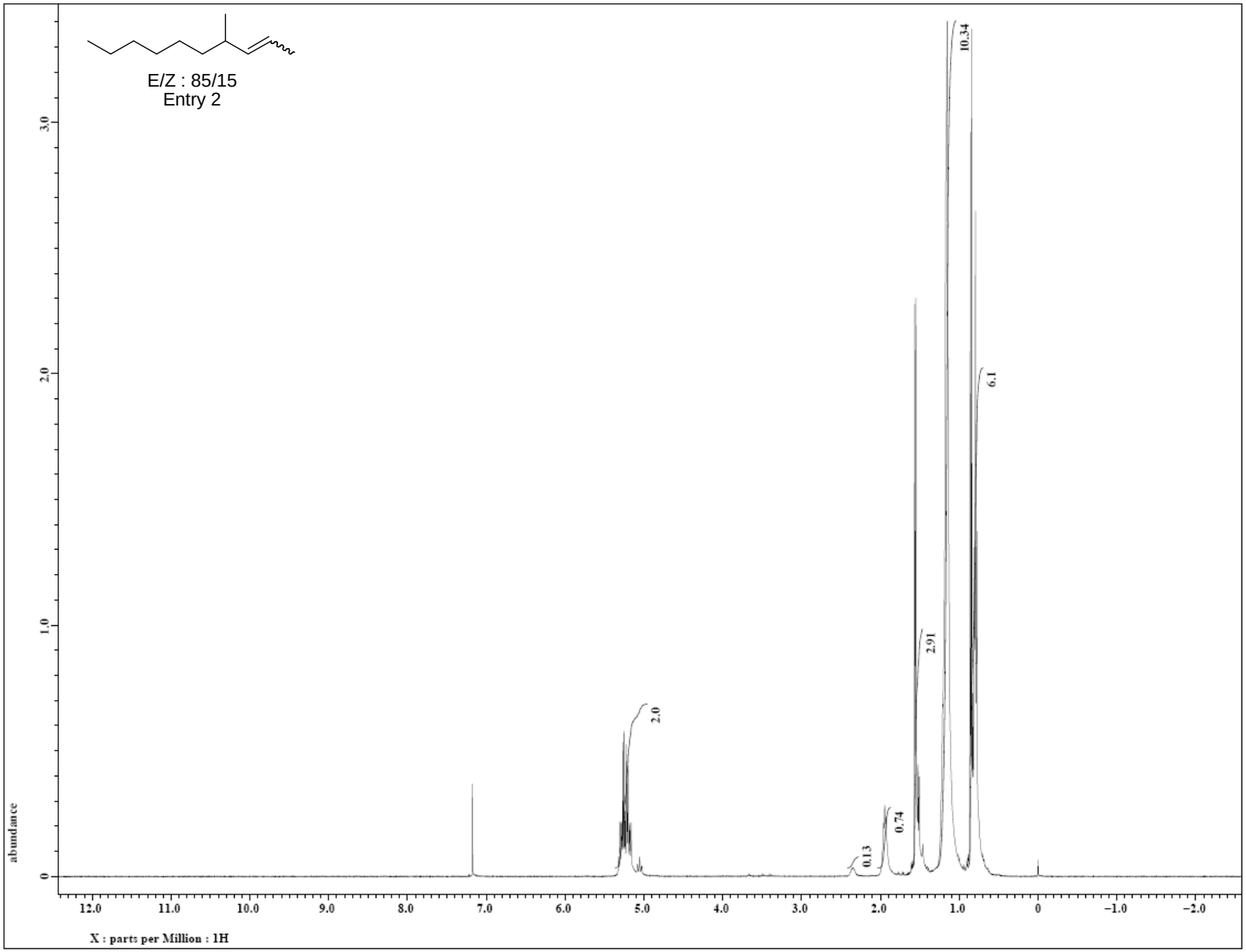


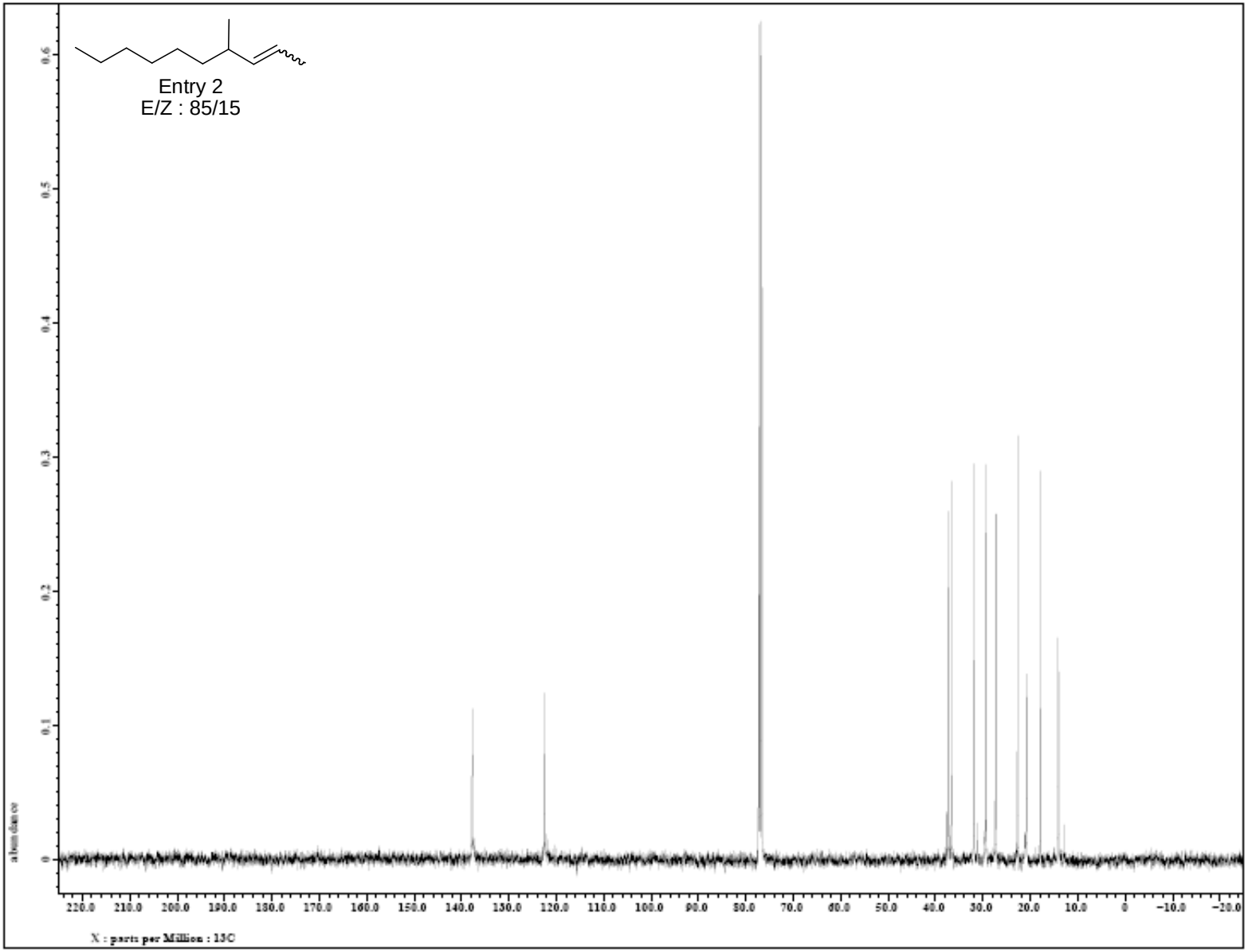
D-2500 00/07/00 06:25

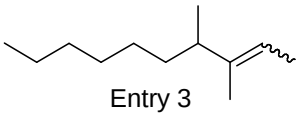
METHOD: SM TAG: 23 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
2	16.11	242	15.224	BB	**Z- Isomer**
3	16.26	1345	84.776	BB	**E- Isomer**
TOTAL		1587	100.000		

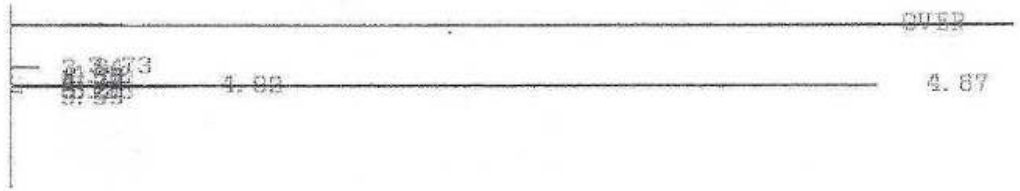

E/Z : 85/15
Entry 2
abundance
X : parts per Million : 1H
0.13
0.74
2.91
2.0
10.34
6.1


Entry 2
E/Z : 85/15
X : parts per Million : 13C

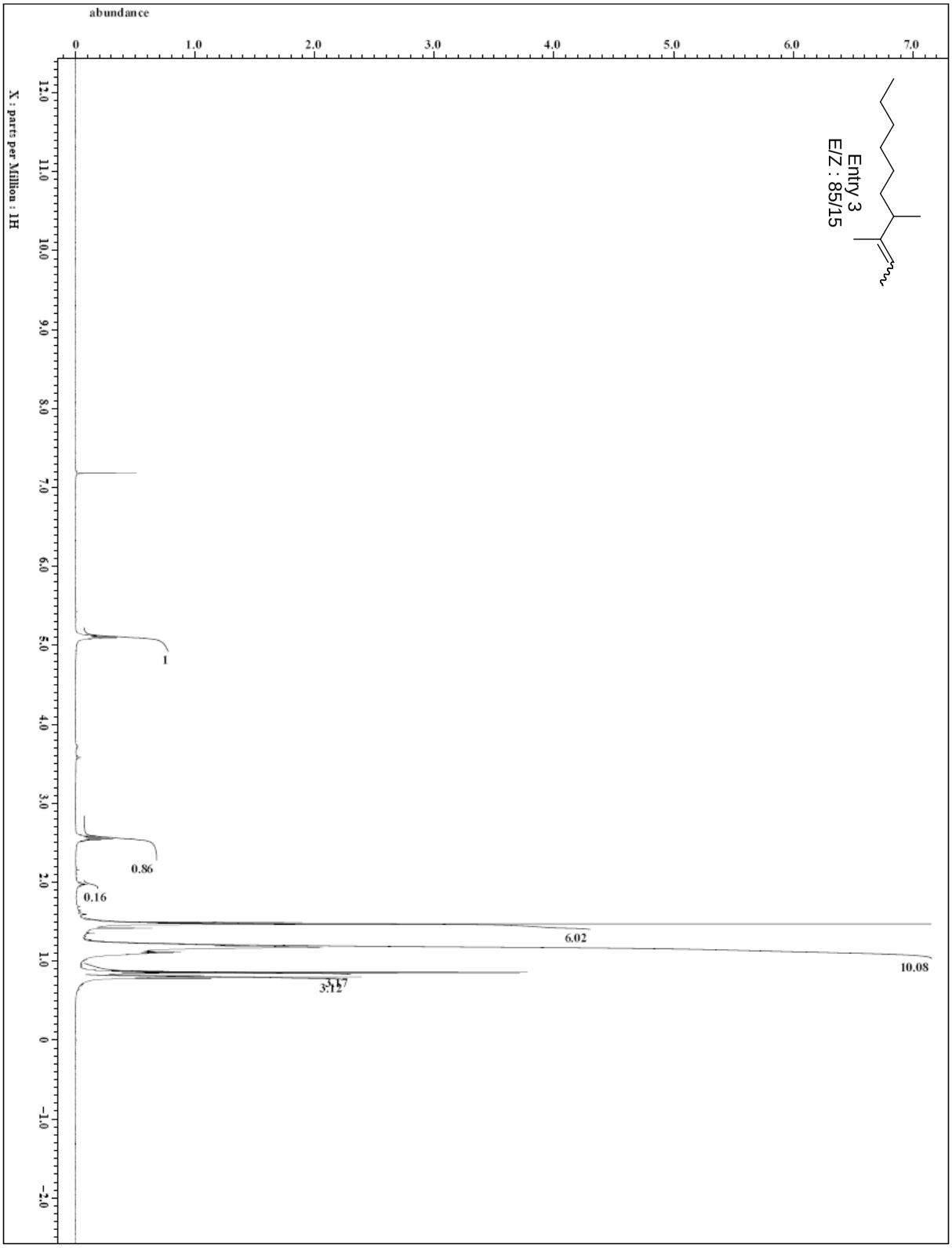


Entry 3
E/Z : 85/15

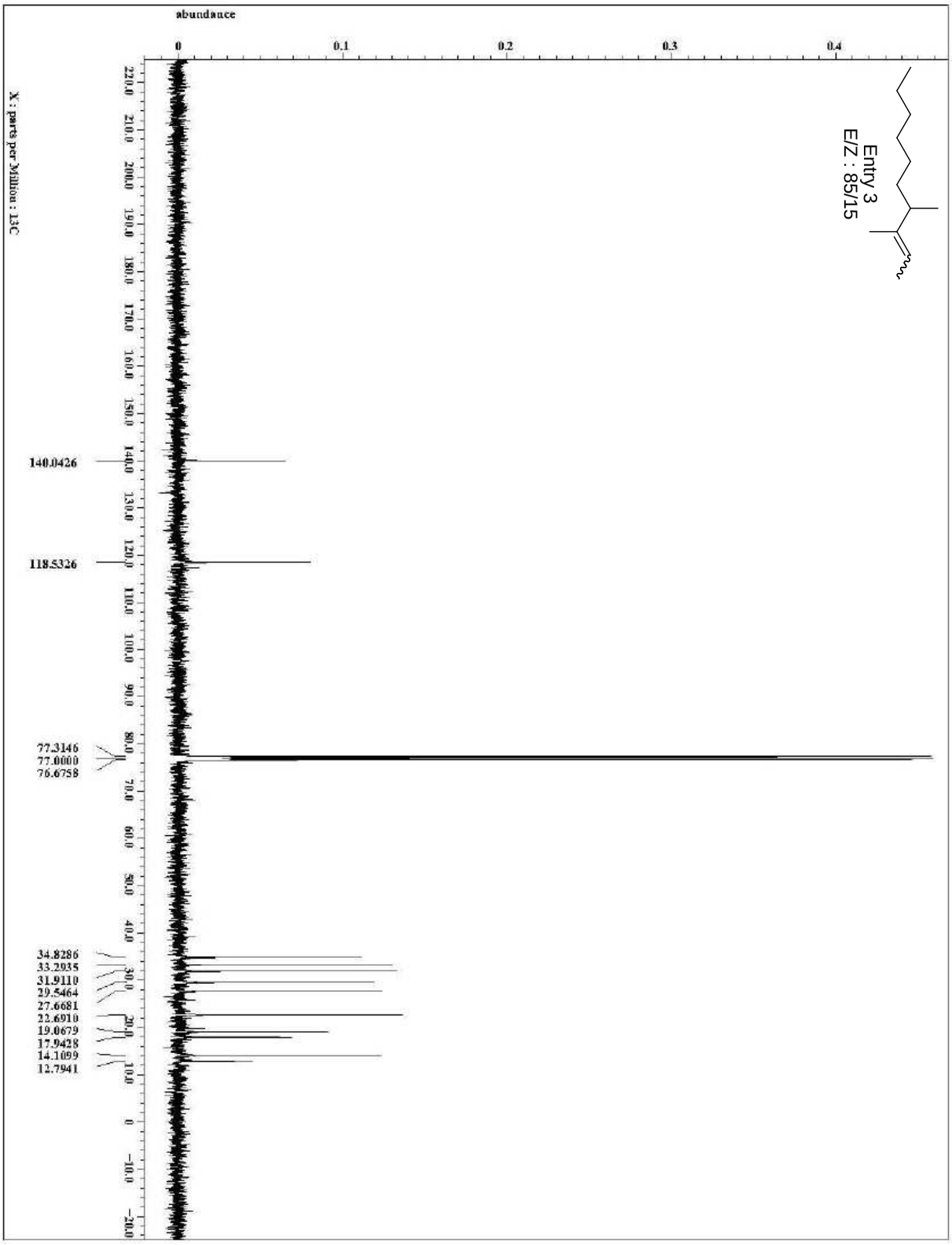
```
FILE 1     SYS 1        SEQ     3
CH.1<A>    C.S  2.50    ATT  5   OFFS   0   05/11/07  10:14
```

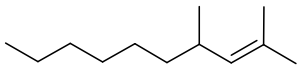



```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/11/07  10:14                  REPORTED: 05/11/07  10:26
SYSTEM   : 1
METHOD   : SM                        OPERATOR: SM
CHANNEL  : 1 <ANALOG>                SEQ     :      3

FILE     : 1 (05/18/07  12:05)
CALC-METHOD: AR/HI%  <AREA>    COMPONENT TBL :   0

  NO.      RT         AREA       CONC   BC
    1    3.73         1087      2.861   BB
    7    4.87        31546     83.018   BV   E-Isomer
    8    4.92         5388     14.121   VB   Z- Isomer
TOTAL
                      37999    100.000
```


abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
Entry 3
E/Z : 85/15
1
0.86
0.16
6.02
10.08
3.12
3.17

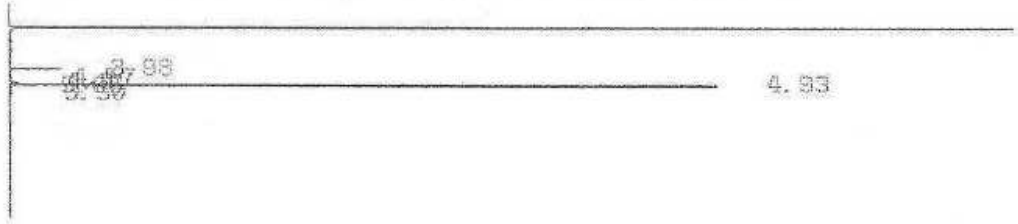
Équation 1


abundance
0
0.1
0.2
0.3
0.4
X : parts per Million : 13C
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
140.0426
118.5326
77.3146
77.0000
76.6758
34.8286
33.2935
31.9110
29.5464
27.6681
22.6910
19.0679
17.9428
14.1099
12.7941
Entry 3
E/Z : 85/15

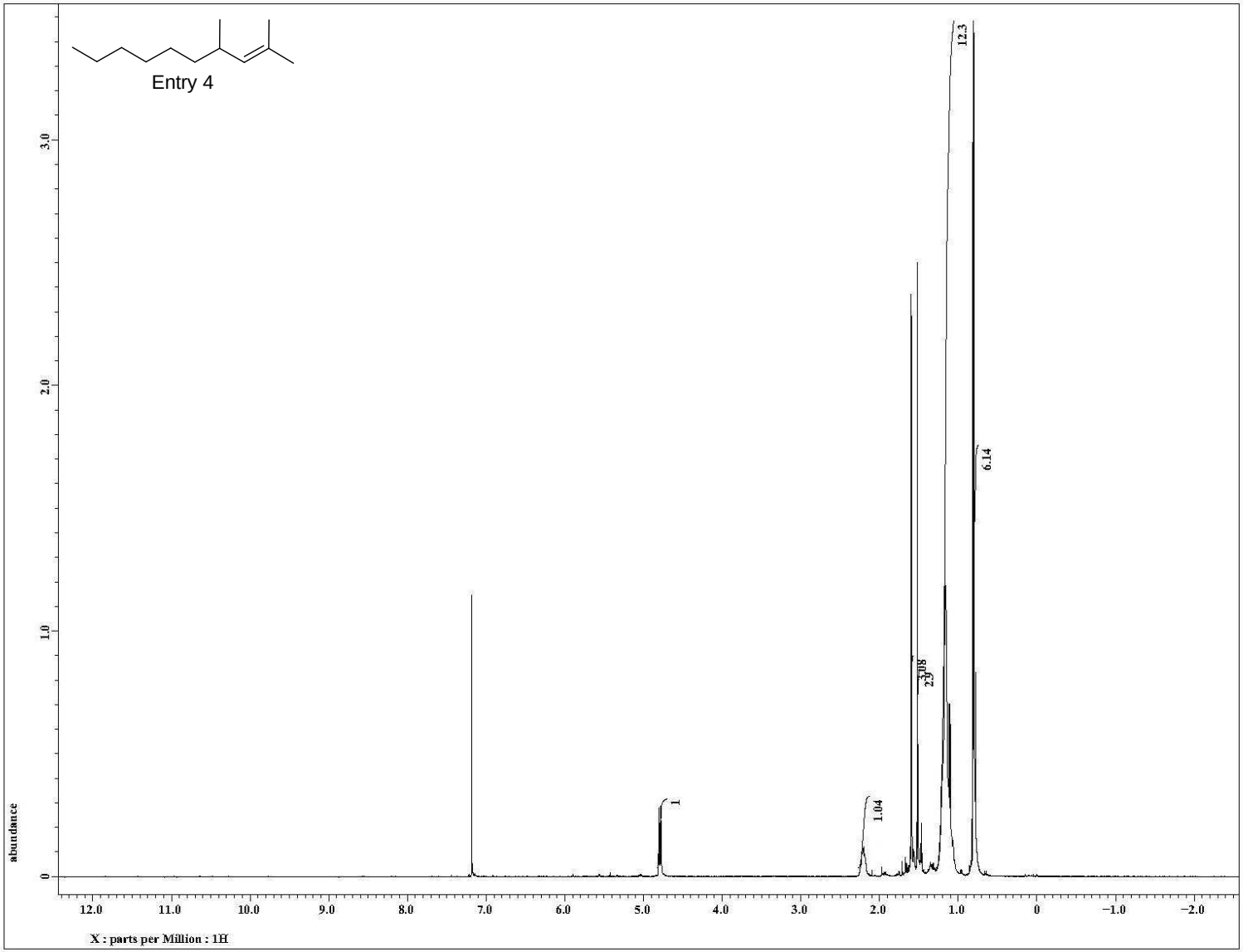


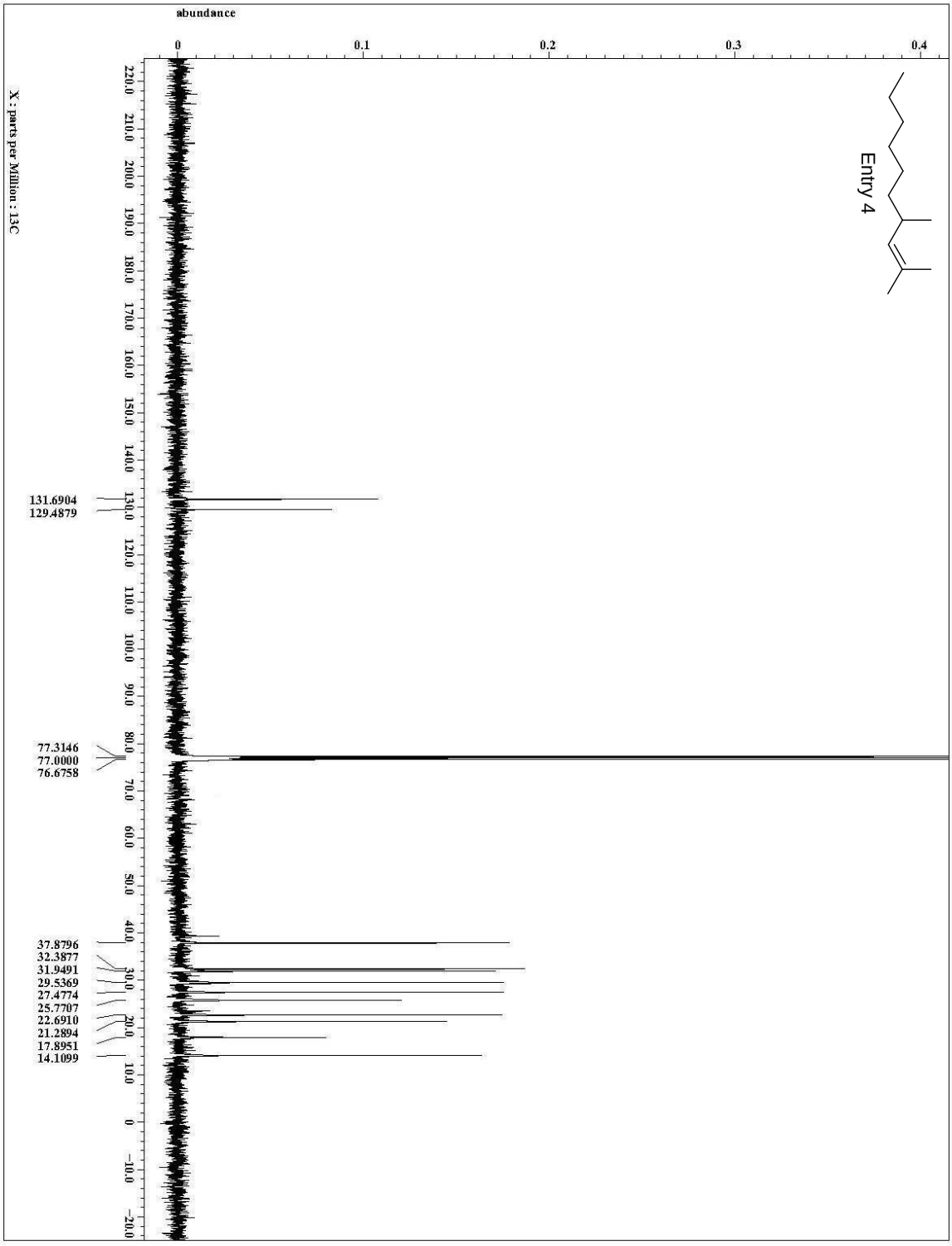
Entry 4

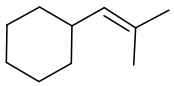
```
FILE 1     SYS 1        SEQ      4
CH.1<A>    C.S  2.50    ATT  5   OFFS    0   05/11/07  11:08
```




```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/11/07  11:08                  REPORTED: 05/11/07  11:34
SYSTEM   : 1
METHOD   : SM                    OPERATOR: SM
CHANNEL  : 1 <ANALOG>            SEQ     :      4

FILE     : 1 (05/10/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC  BC
    2   3.98         974      3.510  BB
    5   4.93       26775     96.490  BB
TOTAL
                   27749    100.000
```

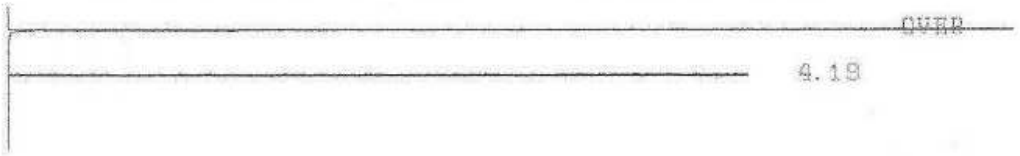

Entry 4
abundance
X : parts per Million : 1H
1
1.04
2.08
2.39
12.3
6.14


abundance
0
0.1
0.2
0.3
0.4
X : parts per Million : 13C
Entry 4
131.6904
129.4879
77.3146
77.0000
76.6758
37.8796
32.3877
31.9491
29.5369
27.4774
25.7707
22.6910
21.2894
17.8951
14.1099

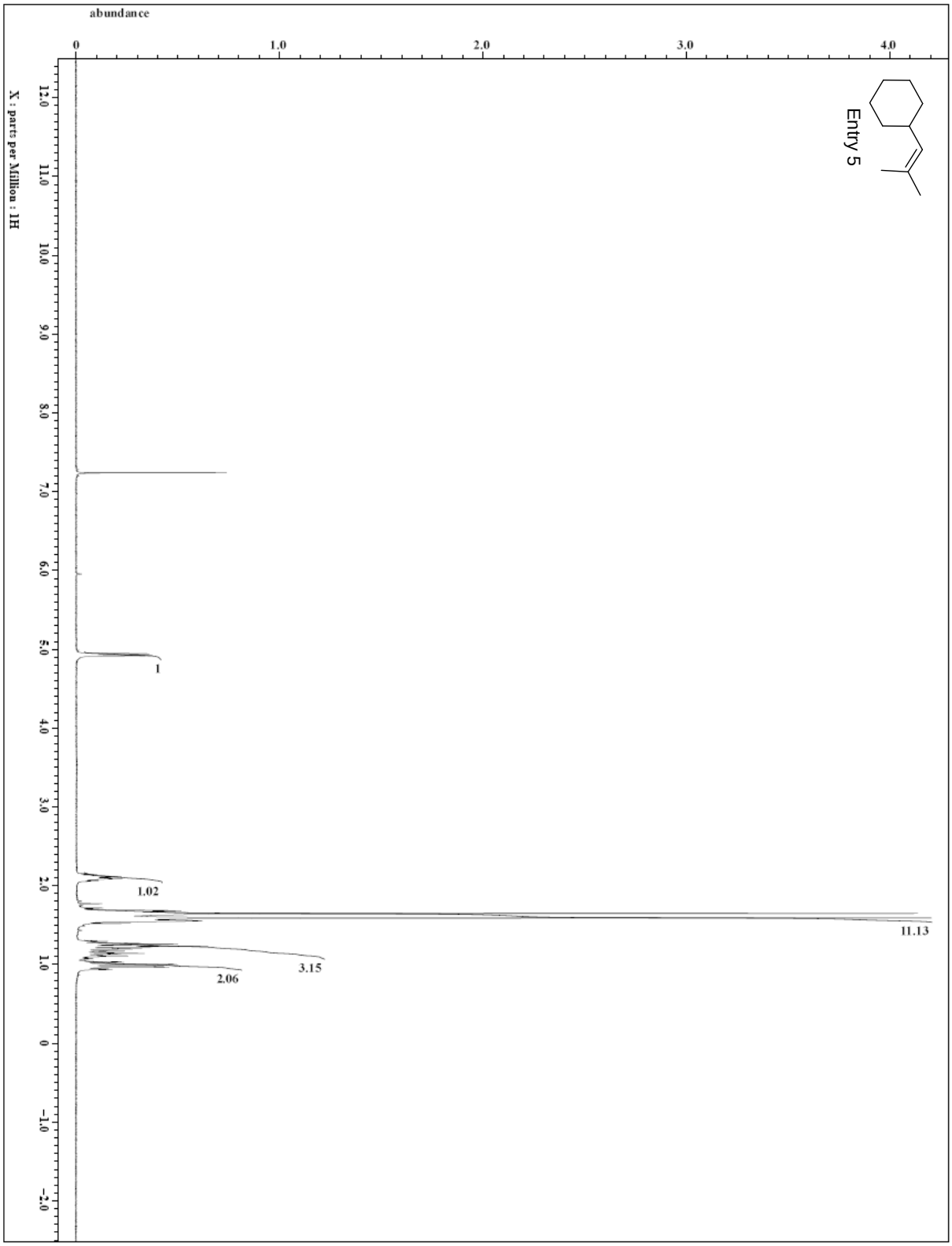


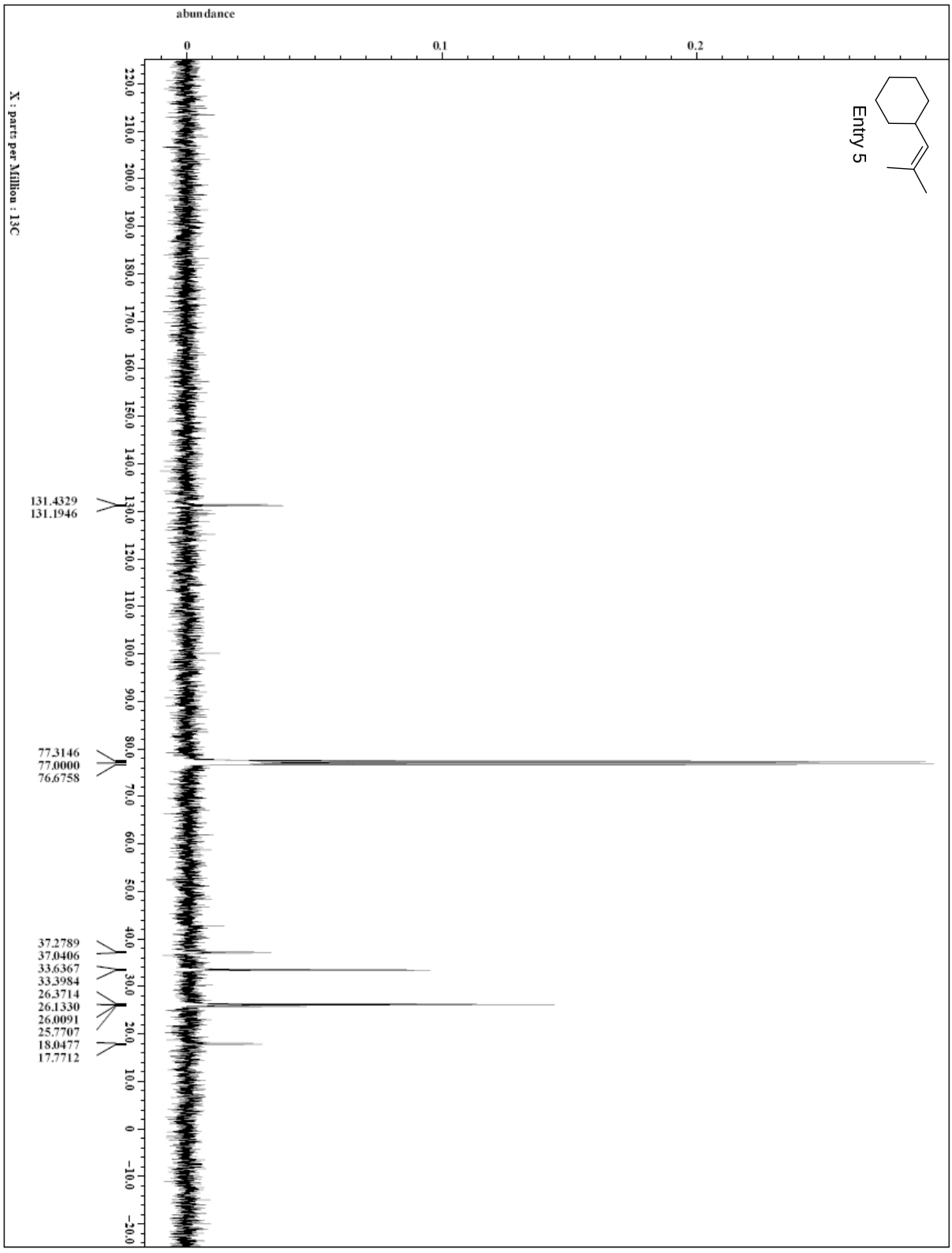
Entry 5

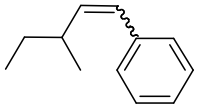
```
FILE 1    SYS 1        SEQ     5
CH.1<A>   C.S  2.50    ATT  5   OFFS   0   05/11/07  11:40
```




```
                    D-7600 INTEGRATOR REPORT

ANALYZED: 05/11/07  11:40                  REPORTED: 05/11/07  11:48
SYSTEM   : 1
METHOD   : SM                   OPERATOR: SM
CHANNEL  : 1 <ANALOG>           SEQ     :      5

FILE     : 1 (05/10/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC  BC
    2    4.19       27260    100.000  BB
TOTAL
                    27260    100.000
```

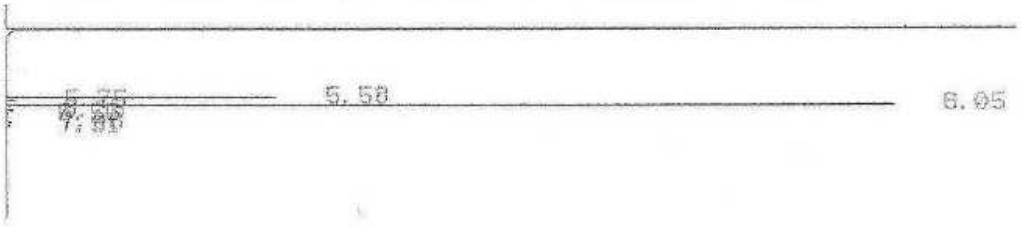

abundance
0
1.0
2.0
3.0
4.0
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
Entry 5
1
1.02
11.13
3.15
2.06


abundance
0
0.1
0.2
X : parts per Million : 13C
Entry 5
131.4329
131.1946
77.3146
77.0000
76.6758
37.2789
37.0406
33.6367
33.3984
26.3714
26.1330
26.0091
25.7707
18.0477
17.7712

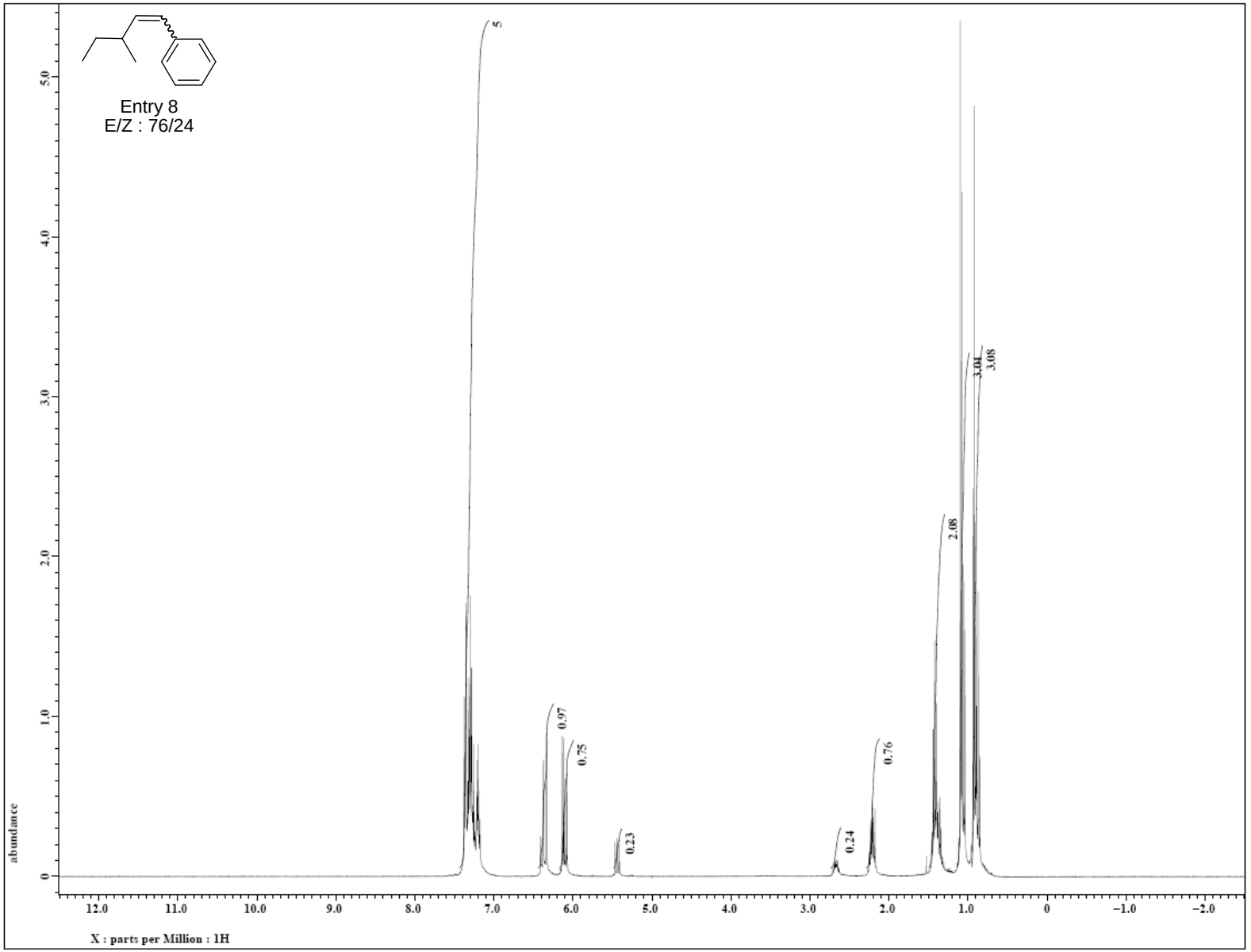


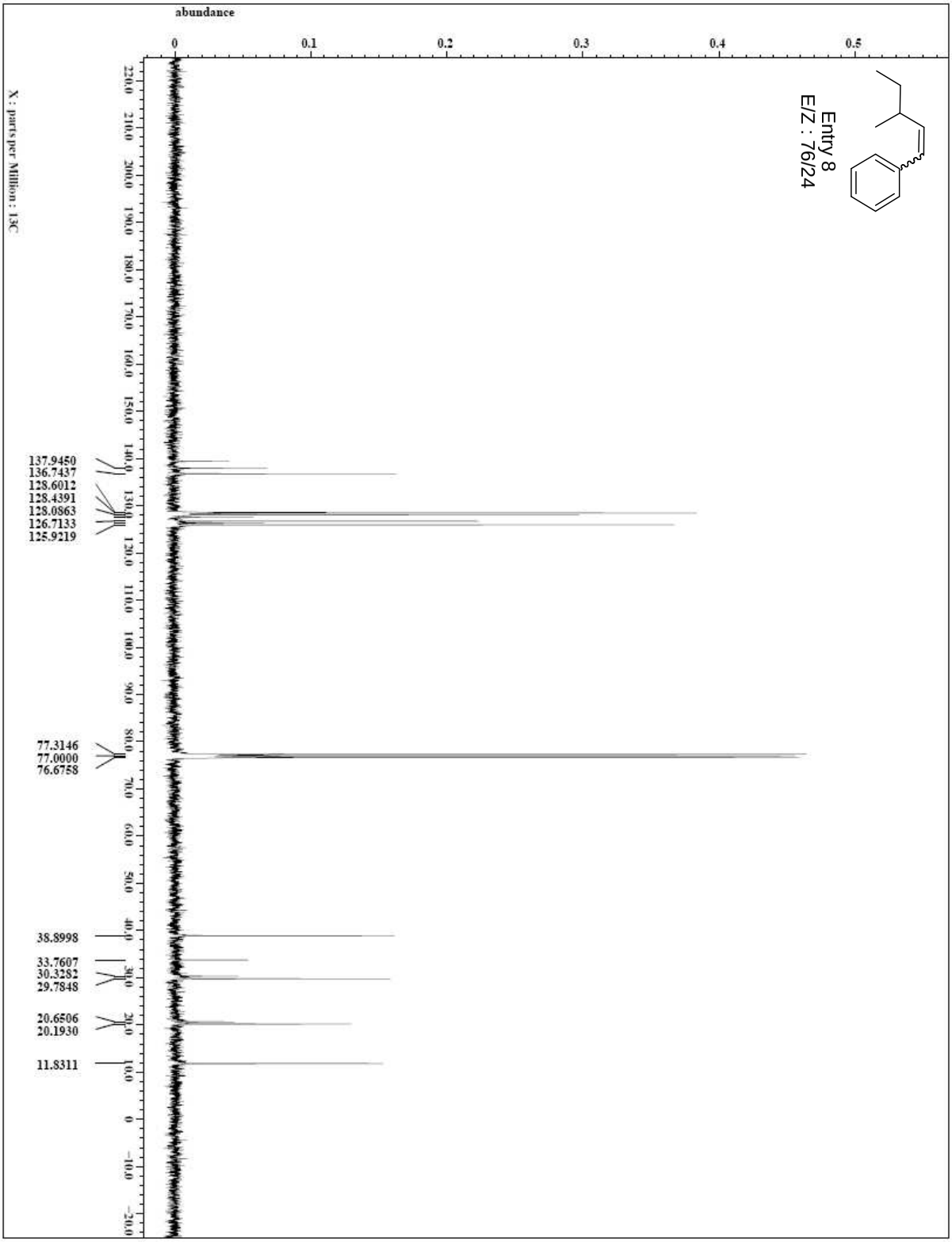
Entry 8
E/Z : 76/24

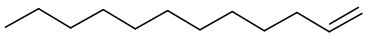
```
FILE 1     SYS 1        SEQ      9
CH.1<A>    C.S  2.50    ATT  4   OFFS    0    05/11/07  14:44
```




```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/11/07  14:44                  REPORTED: 05/11/07  14:59
SYSTEM   : 1
METHOD   : SM                    OPERATOR: SM
CHANNEL  : 1 <ANALOG>            SEQ     :      9

FILE     : 1 (05/10/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC   BC
    1    5.58        4896     23.905   BB   Z- Isomer
    3    6.05       15903     76.095   BB   E- Isomer
TOTAL
                     20800    100.000
```

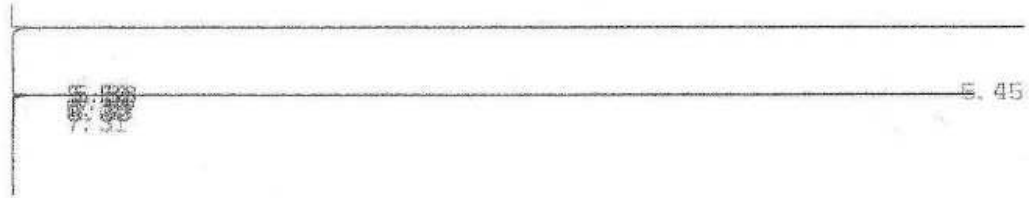

Entry 8
E/Z : 76/24
abundance
X : parts per Million : 1H
0.97
0.75
0.23
0.24
0.76
2.08
3.08


abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
Entry 8
E/Z : 76/24
137.9450
136.7437
128.6012
128.4391
128.0863
126.7133
125.9219
77.3146
77.0000
76.6758
38.8998
33.7607
30.3282
29.7848
20.6506
20.1930
11.8311

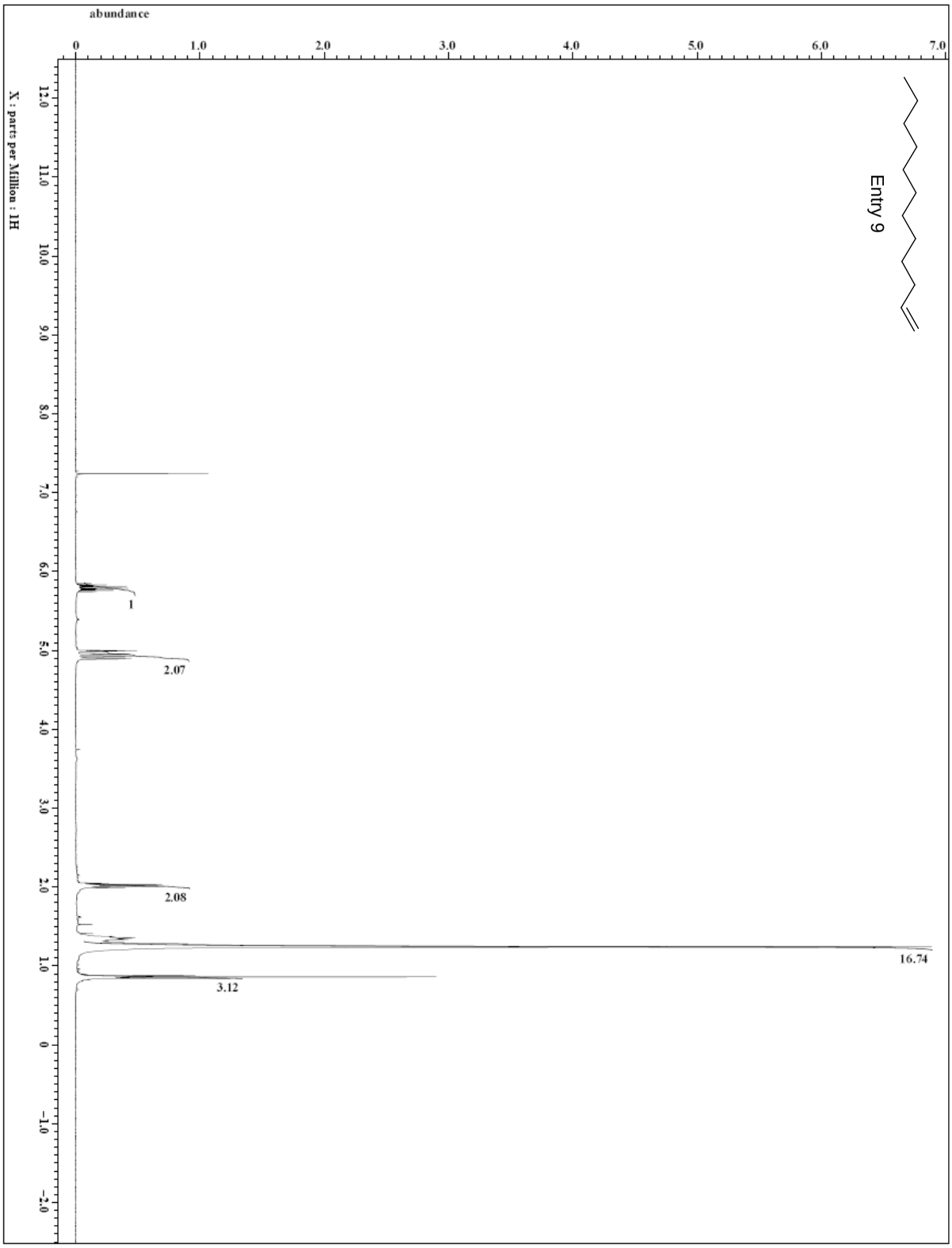


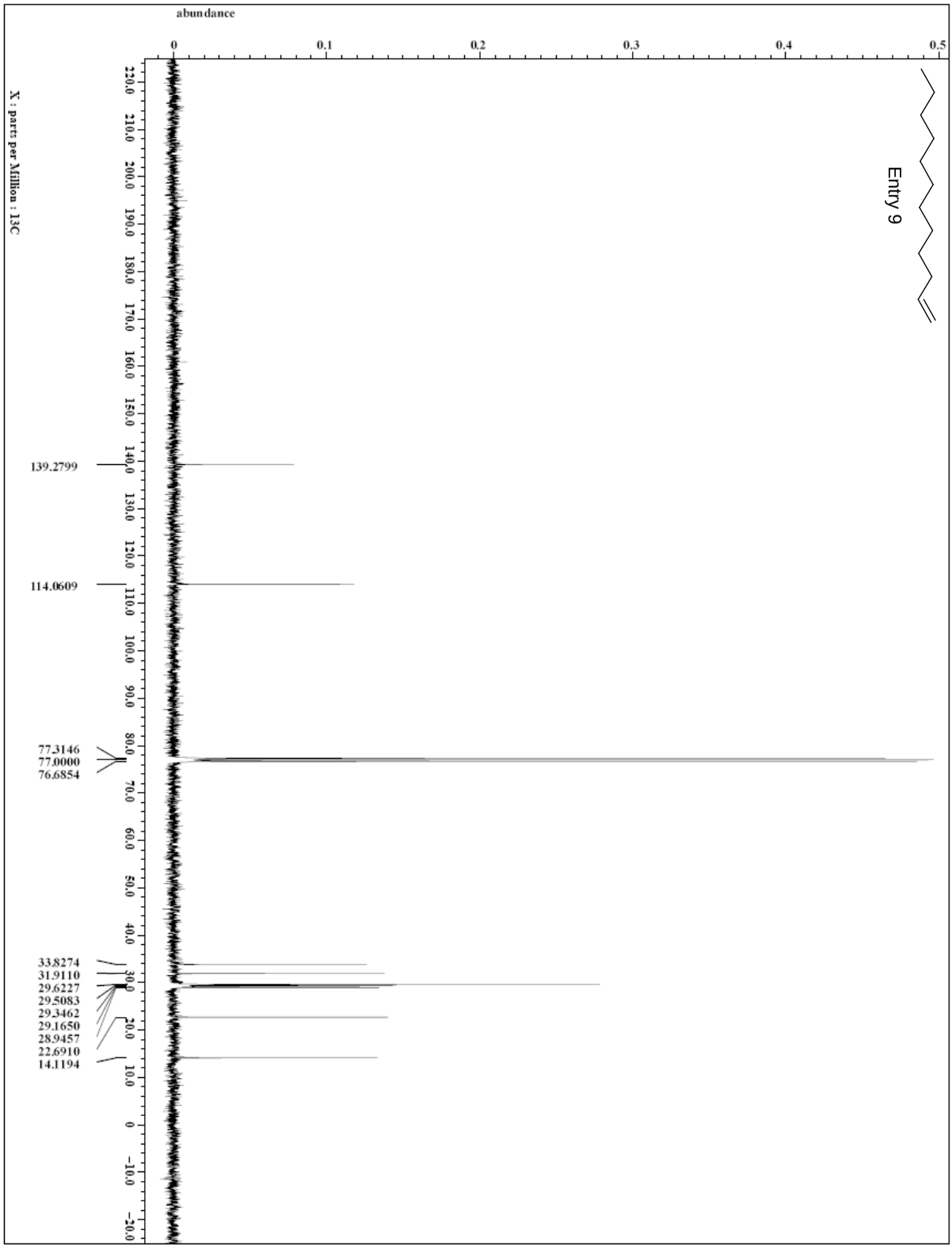
Entry 9

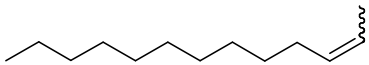
```
FILE 1     SYS 1        SEQ    11
CH.1<A>    C.S  2.50    ATT  4    OFFS    0    05/11/07  15:19
```




```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/11/07  15:18                  REPORTED: 05/11/07  15:33
SYSTEM   : 1
METHOD   : SM                  OPERATOR: SM
CHANNEL  : 1 <ANALOG>          SEQ     :     11

FILE     : 1 (05/10/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC  BC
    3   5.45       16887     97.573  BV
    7   5.96         420      2.427 TBB
TOTAL
                   17307    100.000
```

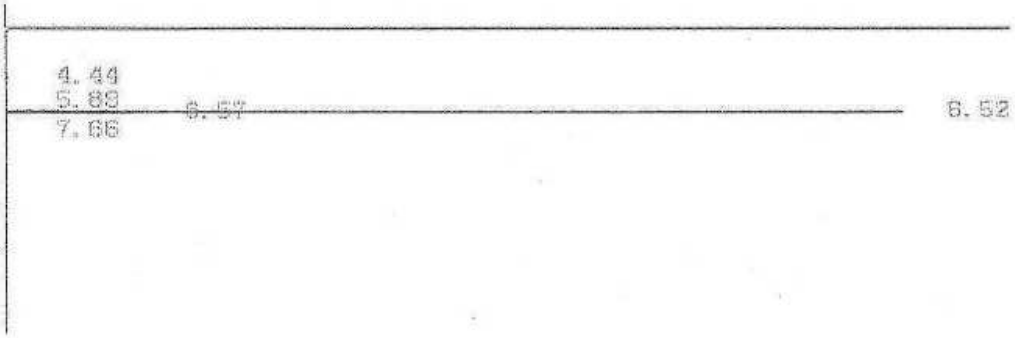

abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
Entry 9
1
2.07
2.08
16.74
3.12


abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
Entry 9
139.2799
114.0609
77.3146
77.0000
76.6854
33.8274
31.9110
29.6227
29.5083
29.3462
29.1650
28.9457
22.6910
14.1194

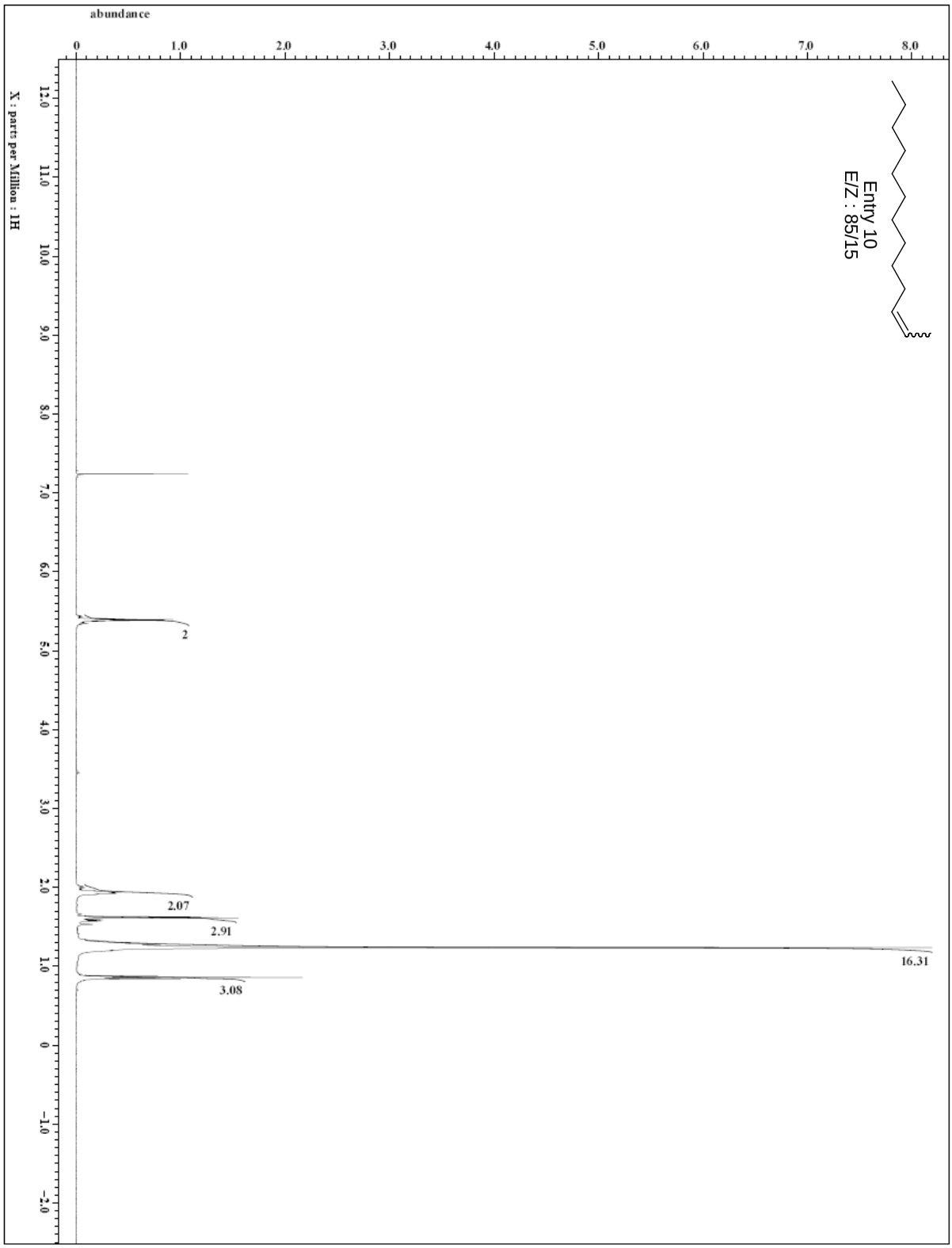


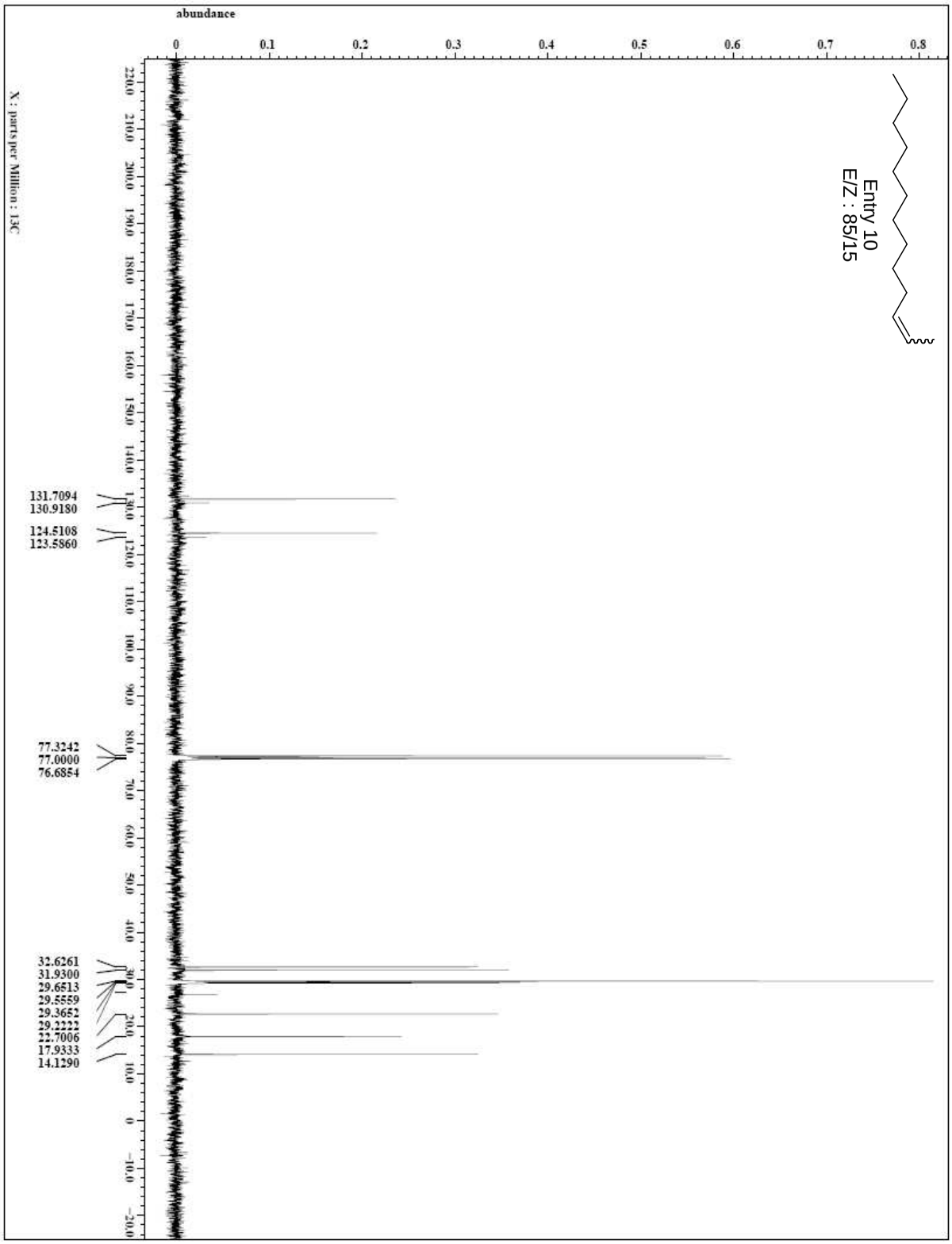
Entry 10
E/Z : 85/15

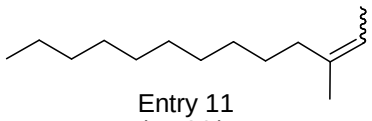
```
FILE 1    SYS 1        SEQ    20
CH.1<A>   C.S  2.50    ATT  5   OFFS   0   05/22/07  14:02
```




```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/22/07  14:02                  REPORTED: 05/22/07  14:24
SYSTEM   : 1
METHOD   : SM                    OPERATOR: SM
CHANNEL  : 1 <ANALOG>            SEQ     :    20

FILE     : 1 (05/18/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC  BC
    3    6.52       31308     85.120  BV
    4    6.57        5488     14.880  VB
TOTAL
                    36877    100.000
```

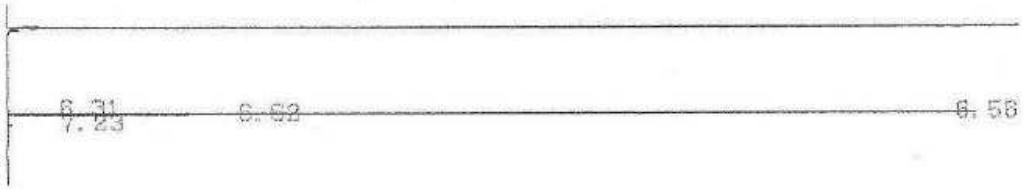

abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
Entry 10
E/Z : 85/15
2
2.07
2.91
16.31
3.08


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
X : parts per Million : 13C
Entry 10
E/Z : 85/15
131.7094
130.9180
124.5108
123.5860
77.3242
77.0000
76.6854
32.6261
31.9300
29.6513
29.5559
29.3652
29.2222
22.7006
17.9333
14.1290

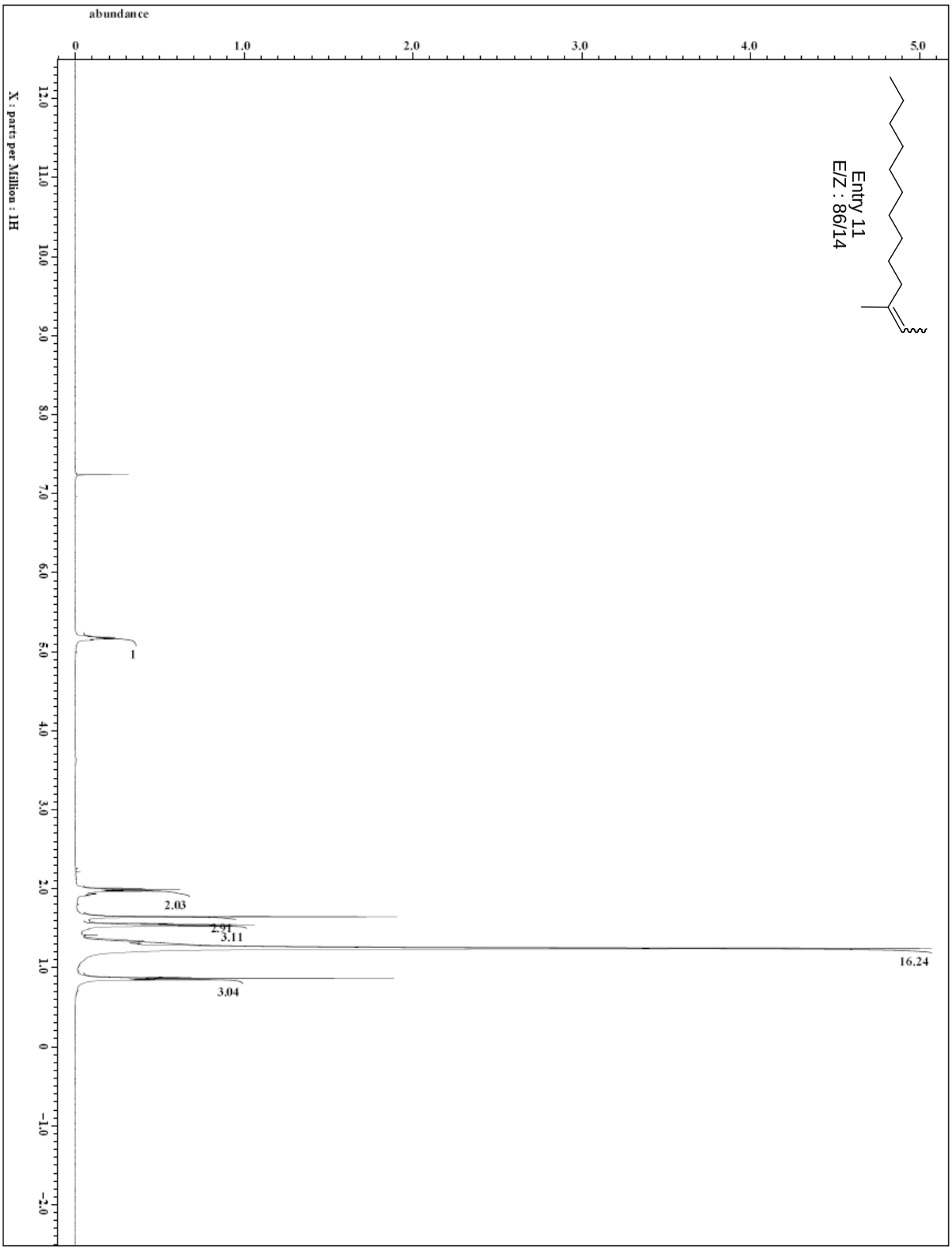


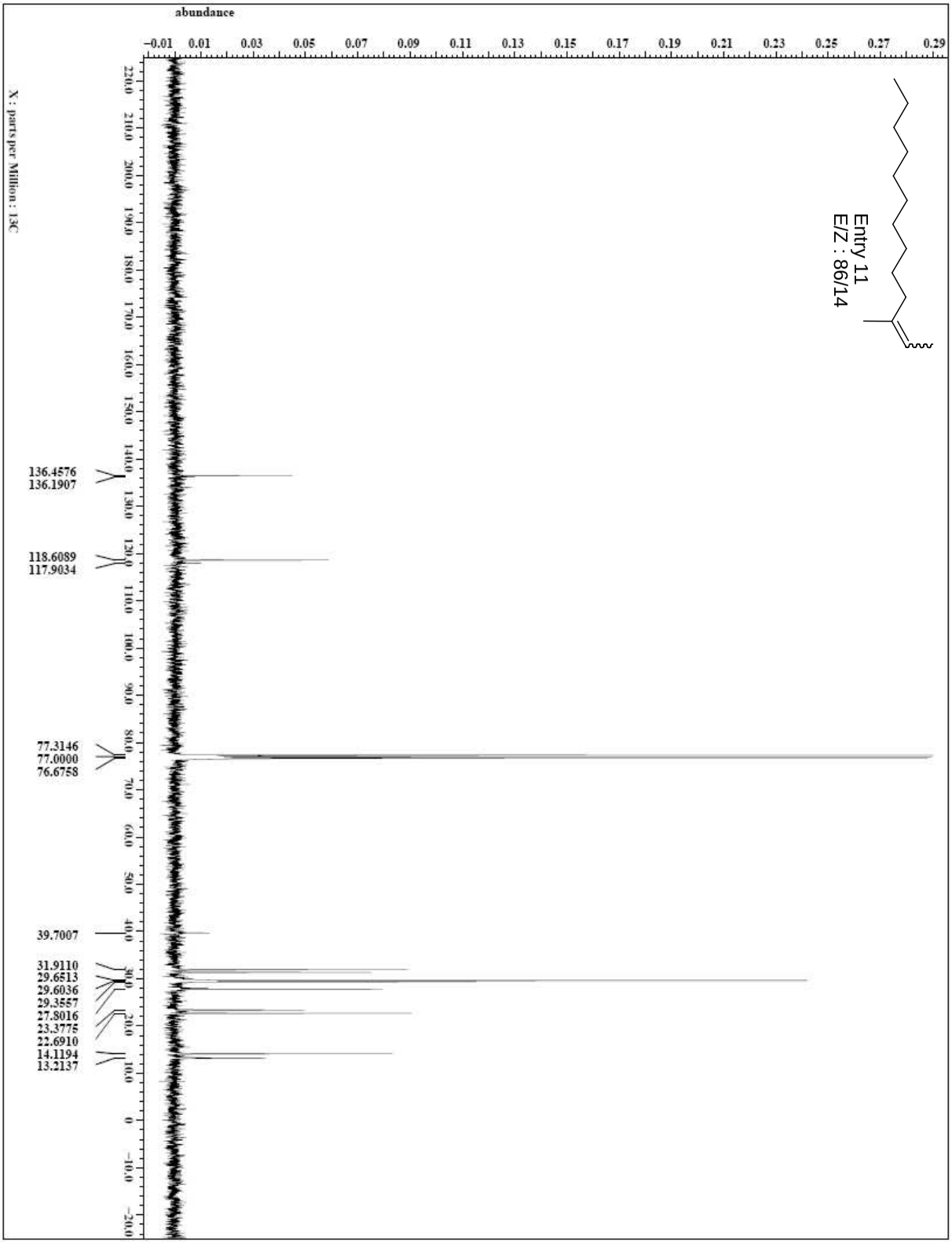
Entry 11
E/Z : 86/14

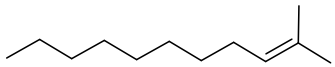
```
FILE 1    SYS 1        SEQ    13
CH.1<A>   C.S  2.50    ATT  4    OFFS   0   05/11/07  16:00
```




```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/11/07  16:00                  REPORTED: 05/11/07  16:11
SYSTEM  : 1
METHOD  : SM                    OPERATOR: SM
CHANNEL : 1 <ANALOG>            SEQ     :     13

FILE    : 1 (05/18/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC  BC
    2   6.56        17205     85.982  BV   E- Isomer
    3   6.62         2708     13.533  VB   Z- Isomer
    4   7.23           97      0.485  BB
TOTAL
                    20010    100.000
```

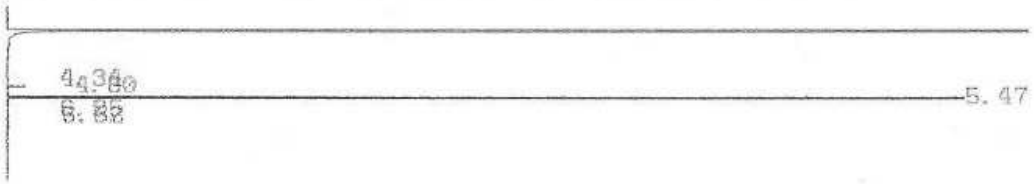

abundance
0
1.0
2.0
3.0
4.0
5.0
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
Entry 11
E/Z : 86/14
1
2.03
2.91
3.11
16.24
3.04


abundance
X : parts per Million : 13C
Entry 11
E/Z : 86/14
136.4576
136.1907
118.6089
117.9034
77.3146
77.0000
76.6758
39.7007
31.9110
29.6513
29.6036
29.3557
27.8016
23.3775
22.6910
14.1194
13.2137

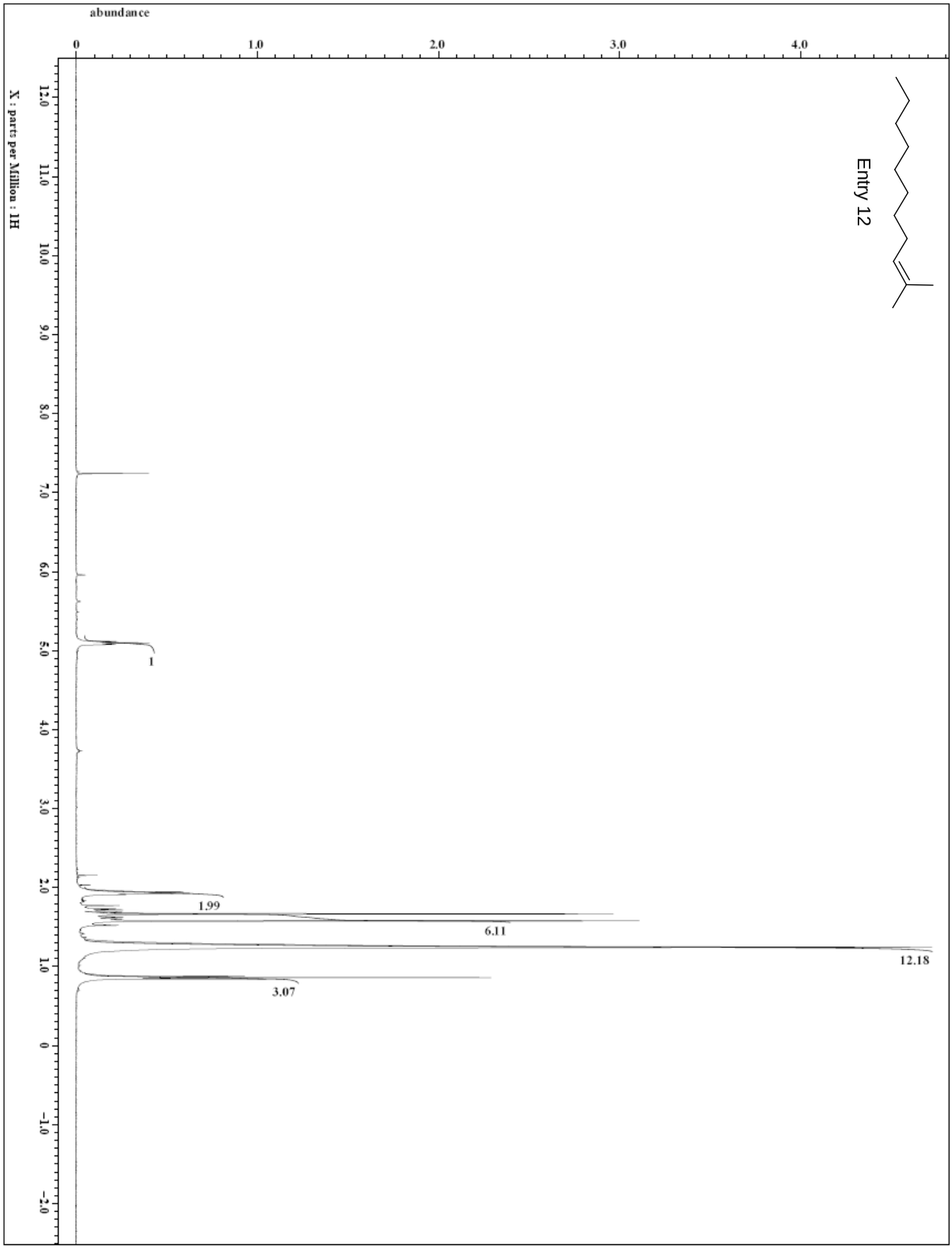


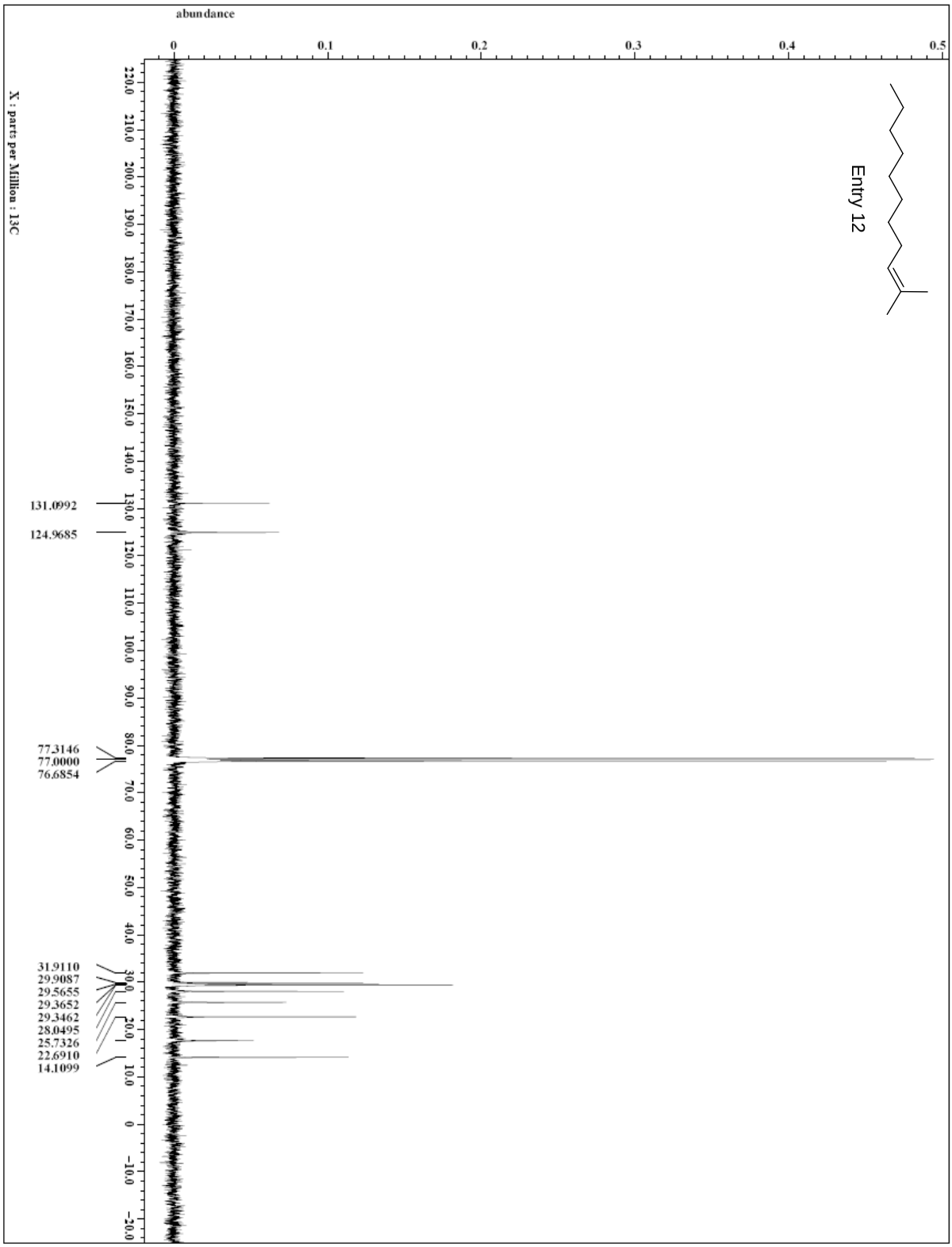
Entry 12

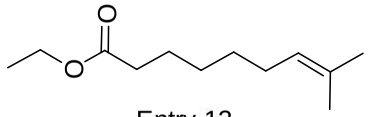
```
FILE 1     SYS 1        SEQ    17
CH.1<A>    C.S  2.50    ATT  3    OFFS    0    05/11/07  16:56
```




```
                    D-7600 INTEGRATOR REPORT

ANALYZED: 05/11/07  16:56                  REPORTED: 05/11/07  17:00
SYSTEM   : 1
METHOD   : SM                    OPERATOR: SM
CHANNEL  : 1 <ANALOG>            SEQ     :     17

FILE     : 1 (05/18/07  12:05)
CALC-METHOD: AR/HI%  <AREA>   COMPONENT TBL :   0

  NO.      RT        AREA       CONC  BC
    3   4.80         191      2.339  BB
    4   5.47        7974     97.661  BB
TOTAL
                    8165    100.000
```

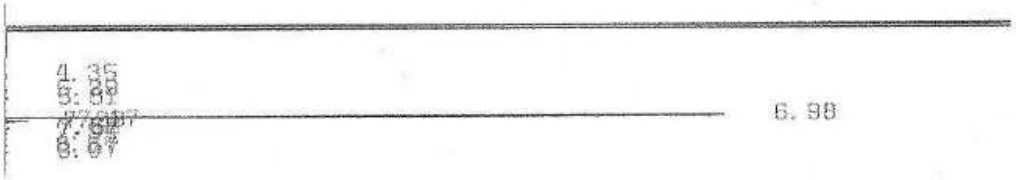

abundance
0
1.0
2.0
3.0
4.0
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
Entry 12
1
1.99
6.11
12.18
3.07


abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
Entry 12
131.0992
124.9685
77.3146
77.0000
76.6854
31.9110
29.9087
29.5655
29.3652
29.3462
28.0495
25.7326
22.6910
14.1099

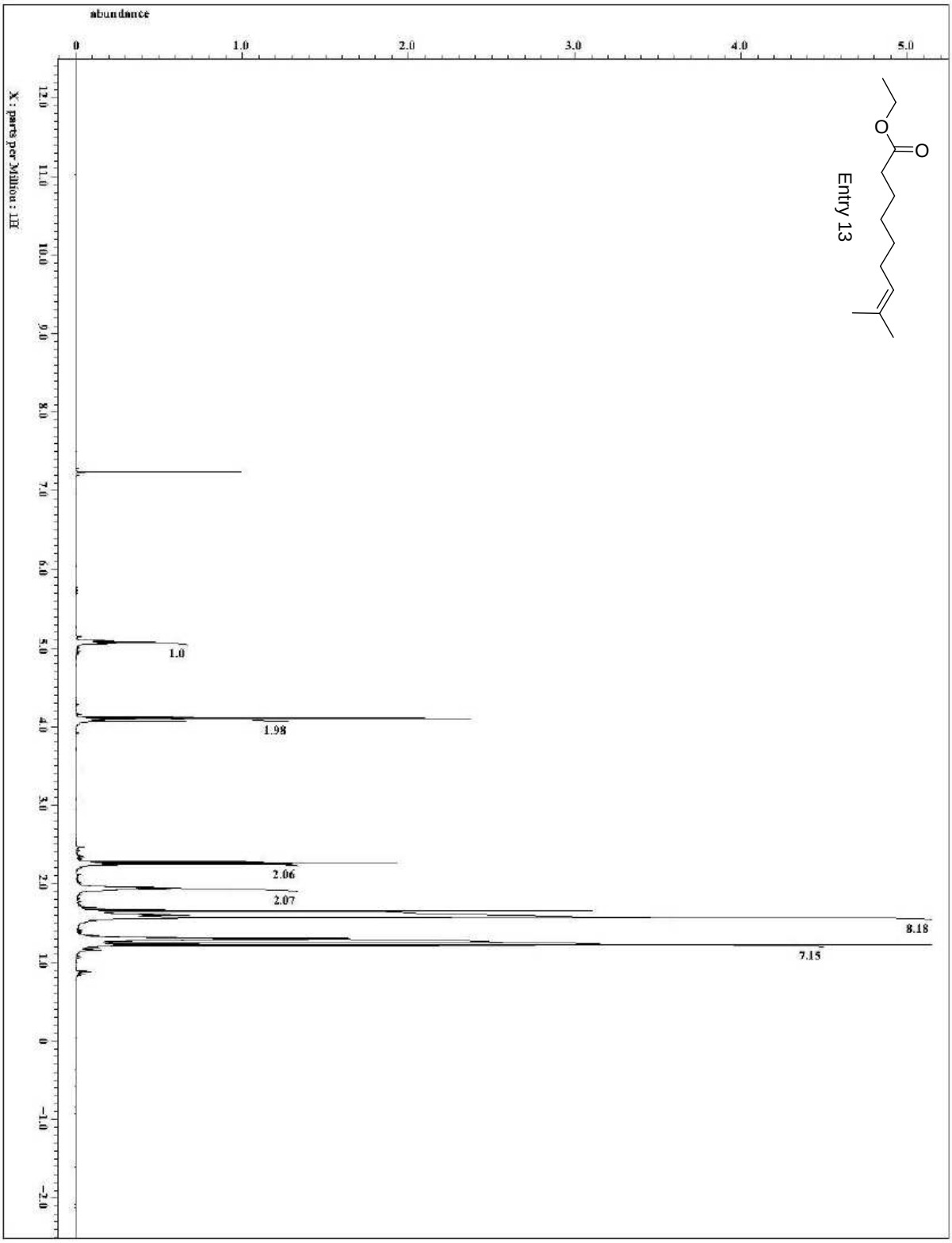


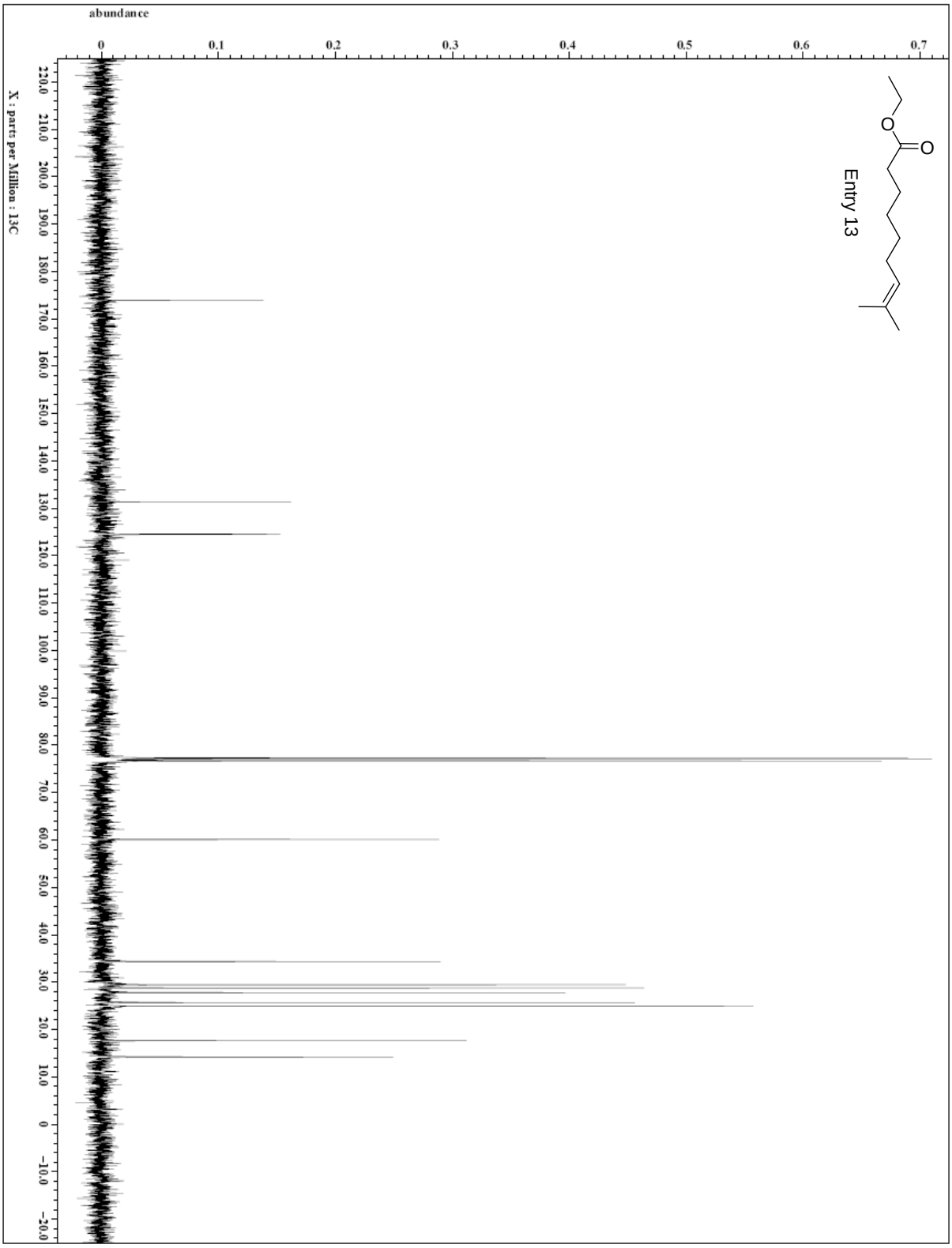
Entry 13

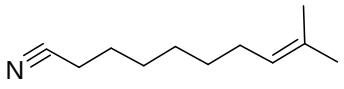
```
FILE 1     SYS 1          SEQ     15
CH.1<A>    C.S  2.50      ATT  5    OFFS    0    05/23/07  19:01
```




```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/23/07  19:01                    REPORTED: 05/23/07  19:12
SYSTEM   : 1
METHOD   : SM                      OPERATOR: SM
CHANNEL  : 1 <ANALOG>              SEQ     :     15

FILE     : 1 (05/18/07  12:05)
CALC-METHOD: AR/HI%  <AREA>    COMPONENT TBL :    0

  NO.      RT         AREA       CONC  BC
    4    6.98        26183     95.438  BB
    5    7.17          852      3.107  BB
    6    7.23          391      1.426  BB
TOTAL
                     27426    100.000
```

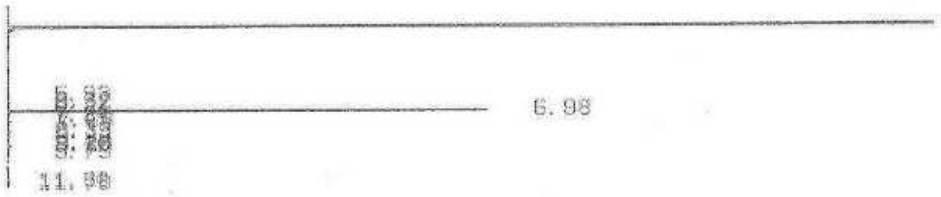

abundance
X : parts per Million : 1H
Entry 13
1.0
1.98
2.06
2.07
8.18
7.15


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
X : parts per Million : 13C
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
O
O
Entry 13

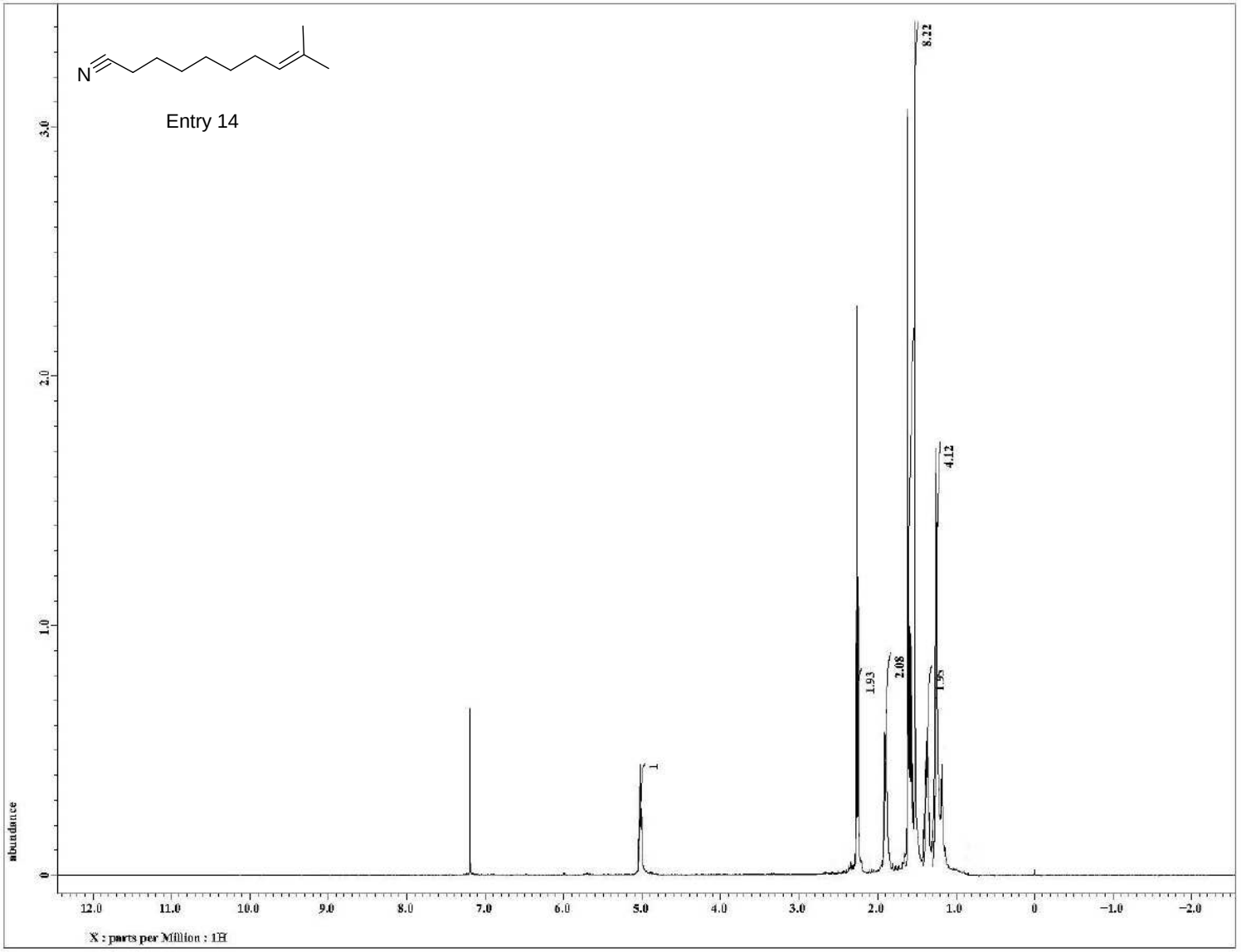


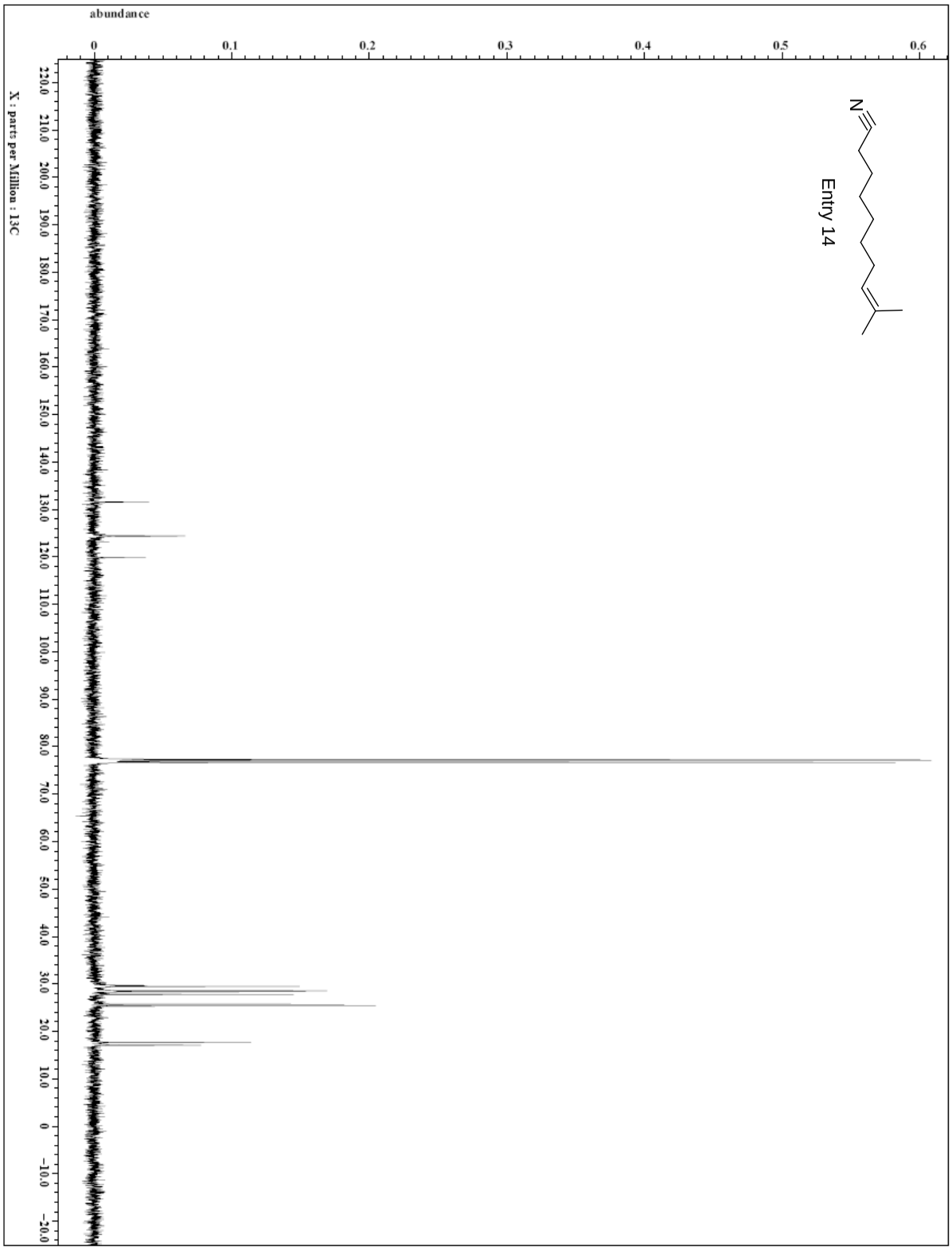
Entry 14

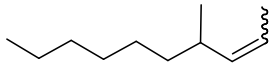
```
FILE 1     SYS 1          SEQ     17
CH. 1<A>   C.S  2.50      ATT  6    OFFS    0    05/23/07  19:34
```



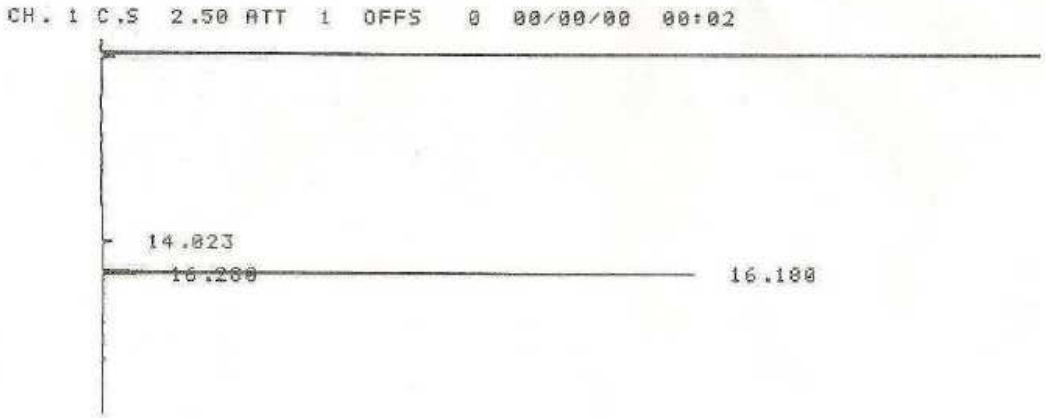

```
                    D-7500 INTEGRATOR REPORT

ANALYZED: 05/23/07  19:34                    REPORTED: 05/23/07  19:49
SYSTEM   : 1
METHOD   : SM                      OPERATOR: SM
CHANNEL  : 1 <ANALOG>              SEQ     :     17

FILE     : 1 (05/18/07  12:05)
CALC-METHOD: AR/HI%  <AREA>    COMPONENT TBL :   0

  NO.      RT        AREA       CONC   BC
    2    6.37         154      0.308   BB
    4    6.98       48814     97.487   BB
    7    7.86         223      0.445   BB
    8    8.25         181      0.361   BV
    9    8.39         143      0.285   VB
   11    9.20         137      0.274   VB
   12    9.28         215      0.429   BB
   14   11.60         168      0.336   BB
   15   11.78         138      0.276   BB
TOTAL
                     50072    100.000
```


N
Entry 14
8.22
4.12
2.08
1.93
1
abundance
X : parts per Million : 1H


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
X : parts per Million : 13C
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
−10.0
−20.0
N
Entry 14



Scheme 1
Z/E : 97/3

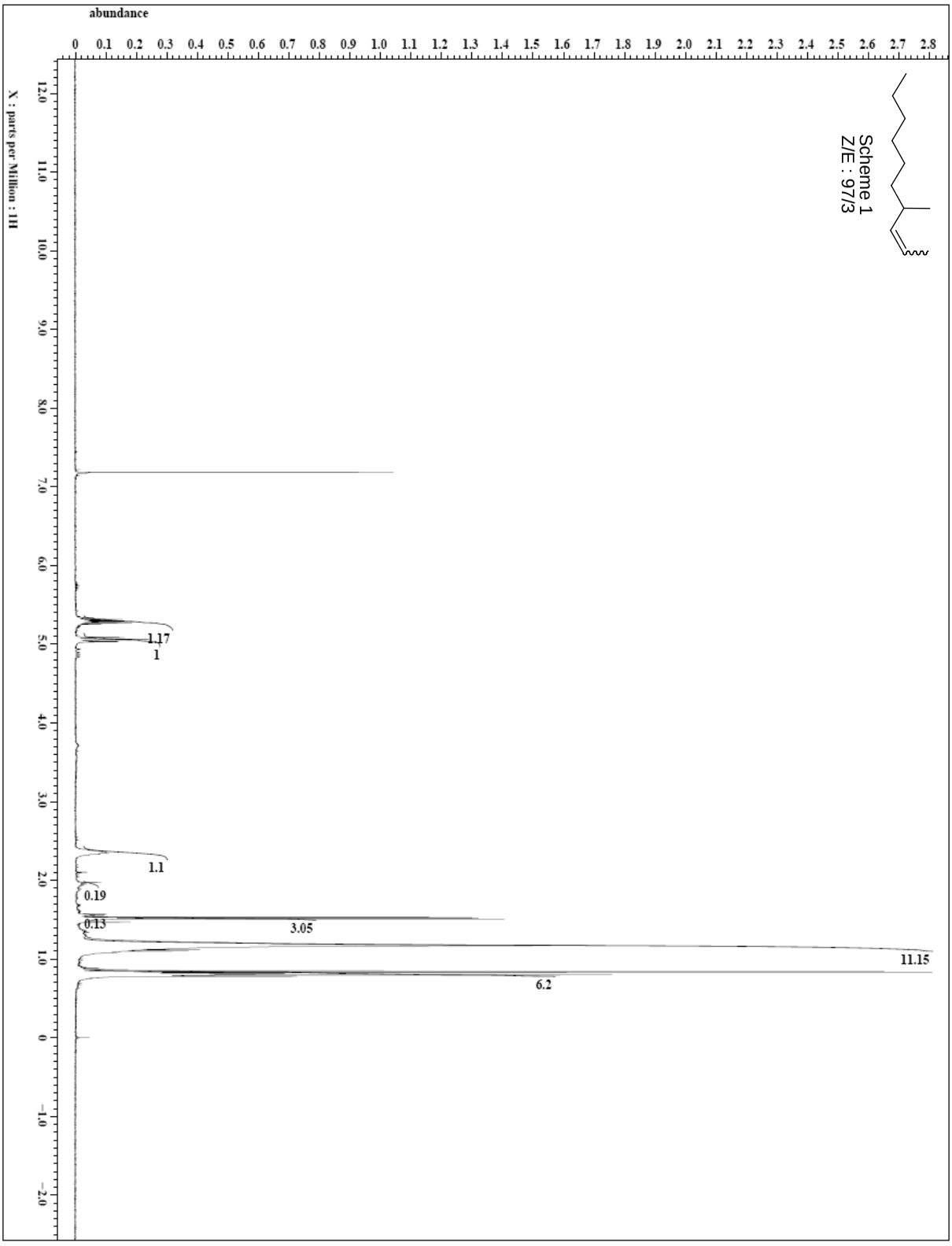


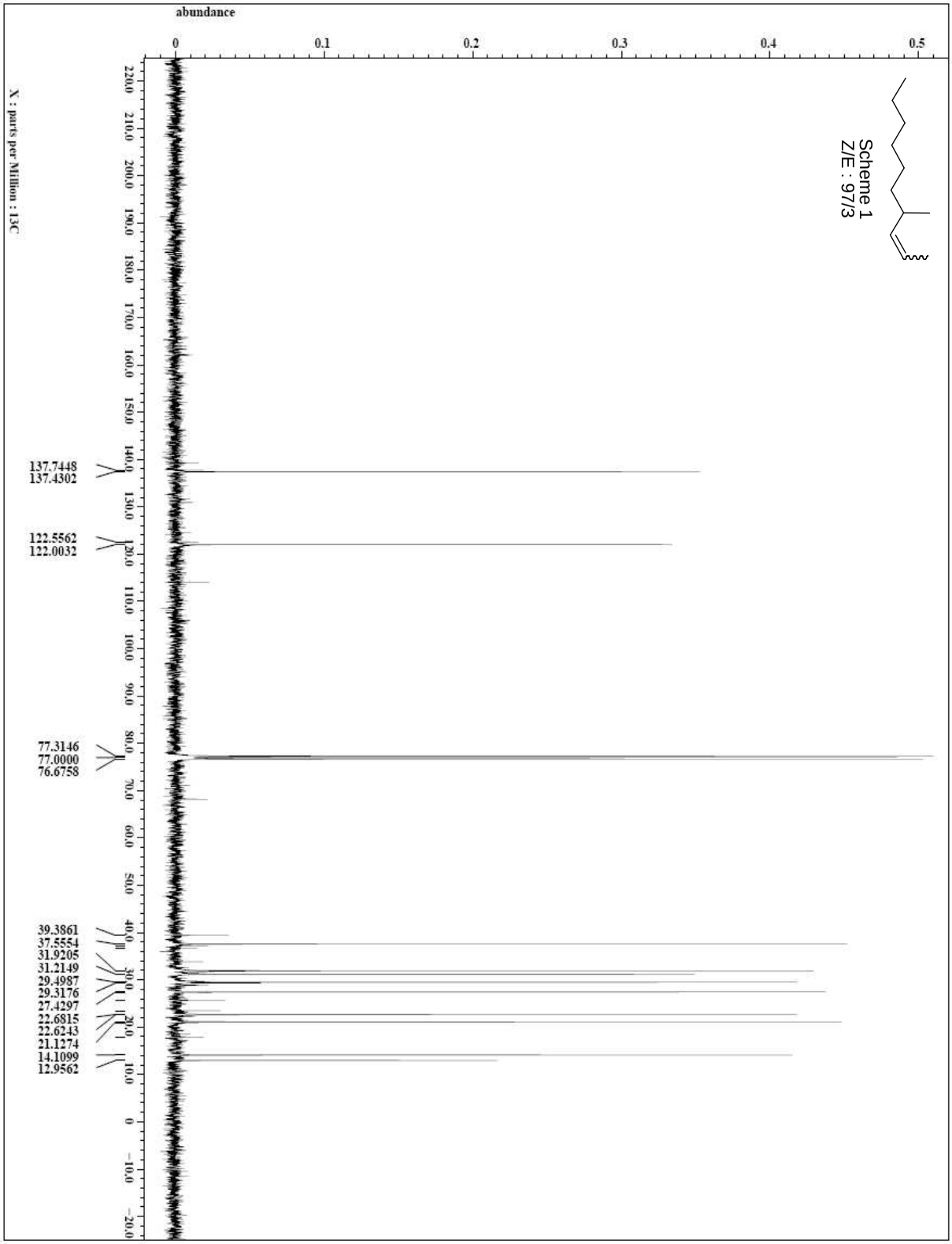
D-2500 00/00/00 00:02

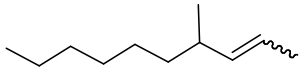
METHOD: TAG: 1 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
2	14.023	106	2.288	BB	
3	16.180	4385	94.654	BB	**Z- Isomer**
4	16.280	142	3.058	BB	**E- Isomer**
TOTAL		4633	100.000		

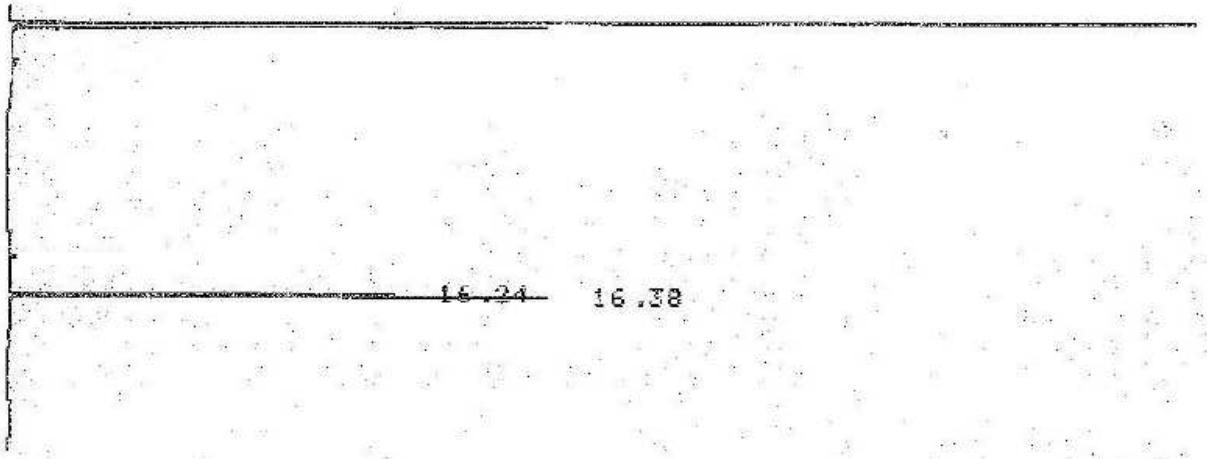

abundance
X : parts per Million : 1H
Scheme 1
Z/E : 97/3
1.17
1
1.1
0.19
0.13
3.05
11.15
6.2


abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
Scheme 1
Z/E : 97/3
137.7448
137.4302
122.5562
122.0032
77.3146
77.0000
76.6758
39.3861
37.5554
31.9205
31.2149
29.4987
29.3176
27.4297
22.6815
22.6243
21.1274
14.1099
12.9562



Authentic Product for Z/E attribution
E/Z : 57/43

CH. 1 C.S 2.50 ATT 0 OFFS 0 00/00/00 19:12



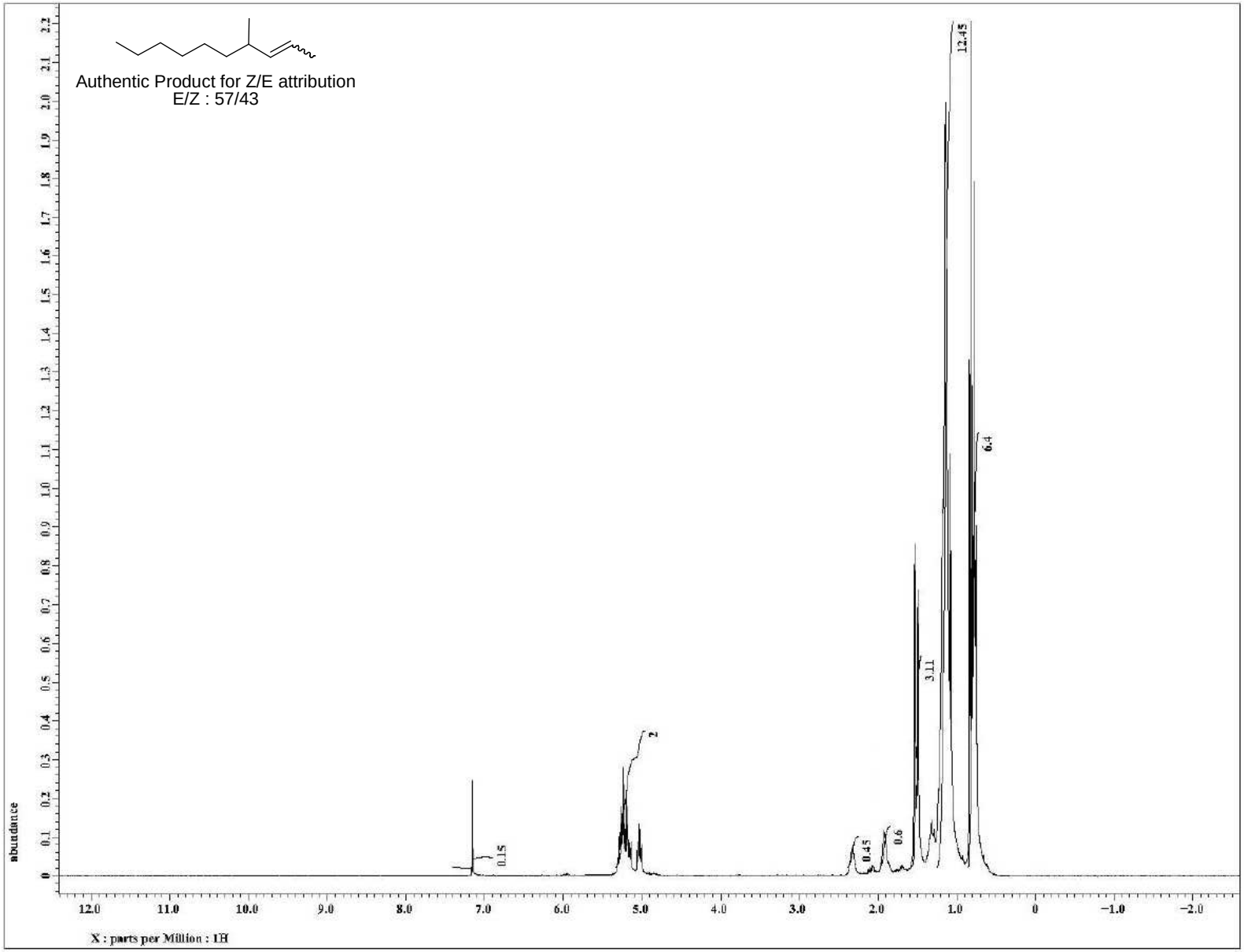
D-2500 00/00/00 19:12

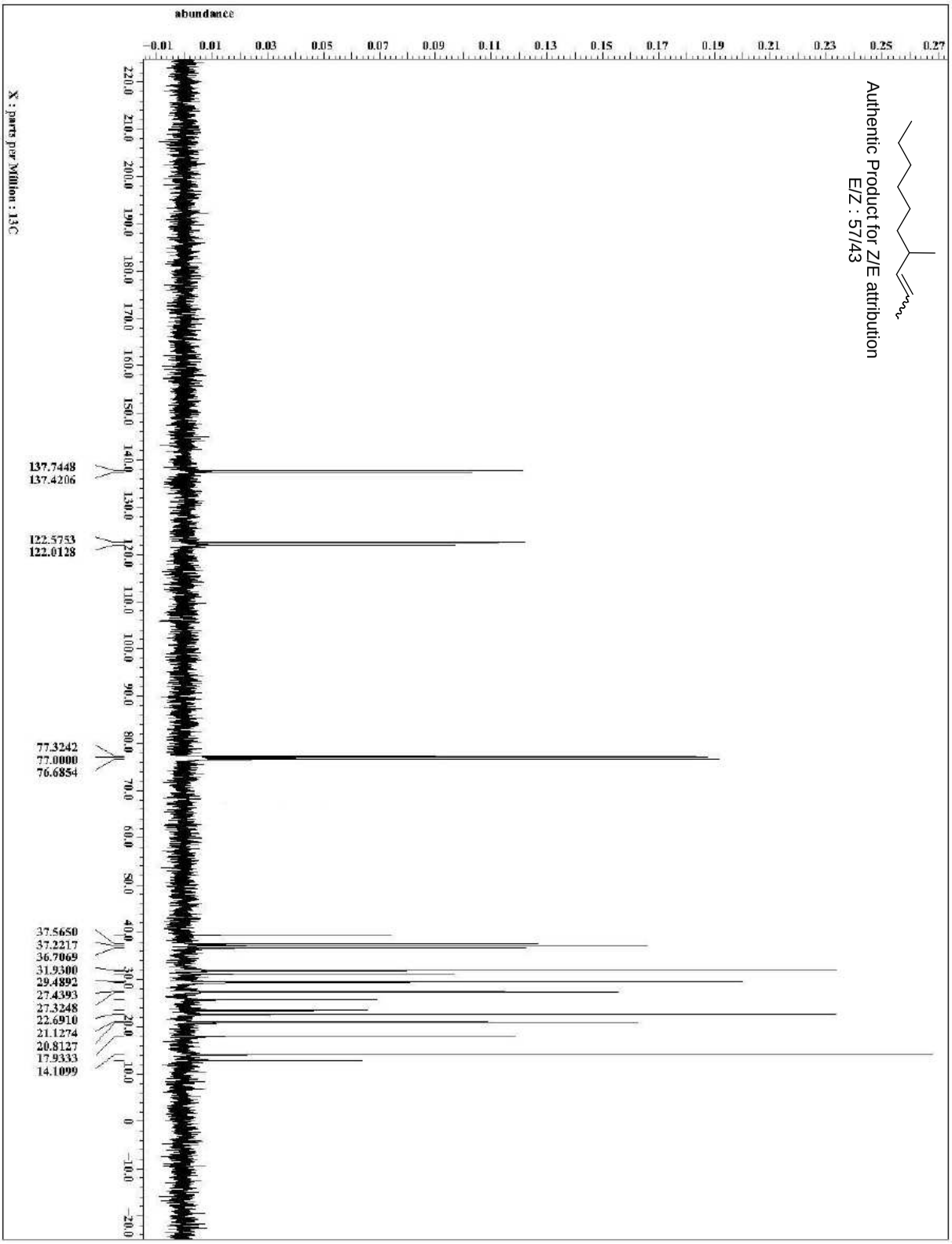
METHOD: SM TAG: 5 CH: 1

FILE: 0 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC	
3	16.24	969	42.781	BB	Z- Isomer
4	16.38	1296	57.219	BB	E- Isomer
TOTAL		2265	100.000		

PEAK REJ : 500


Authentic Product for Z/E attribution
E/Z : 57/43
12.45
6.4
3.11
2
0.6
0.45
0.15
abundance
X : parts per Million : 1H


abundance
X : parts per Million : 13C
Authentic Product for Z/E attribution
E/Z : 57/43
137.7448
137.4206
122.5753
122.0128
77.3242
77.0000
76.6854
37.5650
37.2217
36.7069
31.9300
29.4892
27.4393
27.3248
22.6910
21.1274
20.8127
17.9333
14.1099